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FINAL REMEDIAL INVESTIGATION/FEASIBILITY STUDY SUPPLEMENTAL WORK PLAN NIKE BATTERY SITE PR-58 NCBC DAVISVILLE RI

10/01/2013

STONE ENVIRONMENTAL INC

REMEDIAL INVESTIGATION / FEASIBILITY STUDY SUPPLEMENTAL WORK PLAN

FINAL

NIKE BATTERY SITE PR-58
NORTH KINGSTOWN, RHODE ISLAND

FUDS Project Number: D01RI0029 05
CENAE Project Number: 108346

Contract Number: W912WJ-11-D-0001
Stone Project ID 102305-R
October 2013

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LIST OF ACRONYMS

AHA	Activity Hazard Analyses
AST	above ground storage tanks
APP	Accident Prevention Plan
ARARs	Applicable or relevant and appropriate requirements
bvcA	vinyl chloride reductase
CERCLA	Comprehensive Environmental Response, Compensation and Liability Act
CHA	Carriage Hill Association
COC	contaminants of concern
CSM	conceptual site model
CSS	Contract Compliance Screening
CVOCs	chlorinated volatile organic compounds
DANC	decontamination agent non-corrosive
DCE	dichloroethene
DNAPL	dense non-aqueous phase liquid
DoD	Department of Defense
DPW	Department of Public Works
DQOs	data quality objectives
EDD	electronic data deliverable
EDMS	Environmental Data Management System
fbgs	feet below ground surface
FOC	fraction organic carbon
FOL	field operations leader
FS	Feasibility Study
FSP	Field Sampling Plan
ft	feet/foot
FUDS	Formerly Used Defense Sites
GSA	US General Services Agency
HHRA	Human Health Risk Assessment
IDW	investigation-derived waste
JCO	The Johnson Company
LCS	Laboratory Control Samples
LOD	Limit of detection
LOQ	Limit of quantitation
MCL	maximum contaminant level
MS/MSD	matrix spikes/matrix spike duplicates
NAPL	non-aqueous phase liquid
NAS	Naval Air Station
NAVD	North American Vertical Datum
NCBC	Naval Construction Battalion Center
NCR	non-conformance report
NELAP	National Environmental Laboratory Accreditation Program
PBC	performance-based contract
PCE	tetrachloroethene

PCI	Peabody Clean Industries
PCOCs	potential contaminants of concern
PRG	Preliminary Remediation Goal
Property	former Nike Battery PR-58 property
PWS	*Performance Work Statement*
QAPP	Quality Assurance Project Plan
QA/QC	Quality Assurance/Quality Control
QAO	quality assurance officer
QDC	Quonset Development Corporation
qDHC	Dehalcoccoides spp.
RAO	Remedial Action Objective
RBC	Risk-Based Concentration
RI	Remedial Investigation
RIDEM	Rhode Island Department of Environmental Management
RIEDC	Rhode Island Economic Development Corporation
RSL	regional screening level
SAP	Sampling and Analysis Plan
SEC	Sirrine Environmental Consultants
SEDD	staged electronic data deliverable
SER	Steam Enhanced Recovery
SHE-corrected ORP	standard hydrogen electrode corrected oxidation-reduction potential
SLERA	Screening Level Ecological Risk Assessment
SOP	Standard Operating Procedure
SOW	scope of work
sq. ft	square feet
SSHP	Site Safety and Health
Stone	Stone Environmental, Inc.
TAL	Target Analyte List
TeCA	tetrachloroethane
TCA	trichloroethane
TCE	trichloroethene
tceA	TCE reductase
TIC	tentatively identified compound
µg/Kg	micrograms per Kilogram
USACE	United States Army Corps of Engineers
USEPA	US Environmental Protection Agency
UST	underground storage tank
VC	vinyl chloride
vcrA	vinyl chloride reductase
VOC	volatile organic compound
Work Plan	Feasibility Study Supplemental Work Plan
XML	extensible markup language

1. INTRODUCTION

This Remedial Investigation (RI) / Feasibility Study (FS) Supplemental Work Plan (Work Plan) has been prepared by Stone Environmental, Inc. (Stone) to address planned RI and FS activities at and near the former Nike Battery PR-58 property (the "Property") in North Kingstown, Rhode Island (see Figure 1: Locus Map). Stone Environmental will complete this work under a performance-based contract with the United States Army Corps of Engineers (USACE), New England District, which will be completed as part of the USACE Formerly Used Defense Sites (FUDS) Program. The ultimate goal of the USACE for this Task Order is to propose a remedy (a Proposed Plan), and select the final remedy as part of a Decision Document.

1.1. Purpose of the Work Plan

This Work Plan provides for additional field activities and completion of a RI and FS for the project.

The objectives of the RI/FS field activities are to:

1. Address RI data gaps identified in Stone's *Draft Data Gap Analysis Report*, dated May 25, 2012;
2. Collect data necessary to support an FS, Proposed Plan, and Decision Document, as outlined in the *Performance Work Statement (PWS)*, issued by USACE on November 5, 2010 and subsequently modified on October 31, 2011, December 12, 2011, March 26, 2012, June 15, 2012, July 11, 2012, January 29, 2013, and June 19, 2013.

The objective of the RI is to document the nature and extent of contamination related to the PR-58 Property, and to characterize risk to human health and the environment from such contamination. The objective of the FS is to identify and evaluate the feasibility of remedial alternatives to address the contamination related to the Property, leading to the development of a proposed plan and final selection of a remedy in the decision document.

1.2. Scope of Work

The scope of work for the RI/FS supplemental activities covered by this Work Plan includes the following categories of tasks:

- Project Planning
- Historical Data Analysis
- Field Investigations
- Laboratory Analysis
- Sample Tracking and Data Management
- Evaluation of Remedial Alternatives
- Management and Meetings
- Reporting

1.3. Identification of Participatory and Regulatory Organizations

The PR-58 Property RI and FS are being conducted in accordance with the Comprehensive Environmental Response, Compensation and Liability Act (CERCLA) and the National Contingency Plan to the maximum extent practical, and pursuant to *Environmental Quality - Formerly Used Defense Sites (FUDS) Program Policy* (ER 200-3-1), dated 16 March 2004. Applicable or relevant and appropriate requirements (ARARs)

such as Rhode Island regulations will be incorporated into the RI and FS assessments. It is the policy of the US Environmental Protection Agency (USEPA) and the Department of the Army to ensure that all activities conducted on sites are protective of human health and the environment and to meet (or waive) the substantive provisions of permitting regulations that are ARARs. USEPA has regulatory authority over the adjacent Base Closure and Realignment Commission site and will continue to participate as a stakeholder at the former Nike Battery PR-58. A complete list of participatory and regulatory organizations and their contacts is included in Subsection 10.2.

1.4. Supporting Documents

The development of this Work Plan relied on the current Conceptual Site Model (CSM) included in the *Draft Remedial Investigation (RI) Report* (JCO, 2011) and *Draft Data Gaps Analysis Report* (Stone, 2012). The CSM presents a detailed review of historical uses, past operations, and prior environmental investigations for the Property, along with a summary of Property geology, hydrogeology, contaminant sources and fate and transport processes, potential receptors, and exposure pathways based on current and future property uses. Stone used the CSM to develop a list of data gaps which must be eliminated to complete the RI/FS activities. The CSM is referenced in different sections of this Work Plan and should be consulted for a complete presentation of Property history, prior uses, and contaminant and subsurface conditions.

In support of this Work Plan, the following documents are included as appendices:

- Sampling and Analysis Plan (SAP), prepared in accordance with *Engineer Manual 200-1-3*, which includes the Field Sampling Plan and Quality Assurance Project Plan (QAPP), the latter prepared in accordance with *Uniform Federal Policy for Quality Assurance Project Plans* (DTIC ADA 427785) (Appendix A); and
- The combined Accident Prevention Plan (APP), Site Safety and Health Plan (SSHP), and Activity Hazard Analyses (AHA), prepared in accordance with *Engineer Manual 385-1-1* (Appendix B)

It should be noted that, consistent with USACE policy, Stone has made every effort to eliminate redundancy between the Work Plan and its supporting documents. This effort is intended to streamline USACE and stakeholder review of the Work Plan as well as reduce potential errors and conserve resources. To this end, a number of tables are cross-referenced between documents, primarily between the Work Plan and SAP, rather than being reproduced in their entirety in both documents.

The Work Plan focuses on background information, data gaps and overall sampling rationale, the overall scope of work (SOW), project schedule, and project personnel and their responsibilities. The SAP focuses on detailed descriptions of sampling objectives, locations, frequency, designations, equipment and procedures, and handling and analysis. The QAPP focuses on data quality objectives (DQOs) and the measures in place to document that DQOs are achieved. Finally, the combined APP/SSHP/AHA documents the planning and procedures in place to perform all work safely.

2. SITE BACKGROUND AND SETTING

The following section provides an overview of the background and setting. A detailed review of the background and setting for the Property and its vicinity can be found in the CSM.

2.1. Property Identification

The Property is located in the community of North Kingstown, Rhode Island (see Figure 1: Locus Map). The Property encompasses approximately 39 acres and is bounded by former Perimeter Road to the north, Seabee Avenue to the east, Newcomb Road to the west, and Babcock Road to the south (see Figure 2: Property and Sample Locations).

For ease of reference, locations of monitoring wells installed during previous investigations are provided on Figures 3a and 3b. Historic pore water, surface water, and sediment sampling locations, as well as staff gauge locations, are shown on Figure 2. Historical onsite uses and potential sources of contamination are shown on Figure 4.

The areas to the north and west of the Property are largely residential with minimal, light commercial / industrial land use. To the south of the Property is the Quonset Development Corporation (QDC) commercial development of the former Quonset Point Naval Air Station (NAS) and to the east is the former Davisville Naval Construction Battalion Center (NCBC). The NAS Site and NCBC NPL Site are the focus of their own contamination investigations and remediation planning.

2.2. Property History

Prior to 1942, the Property was a mostly wooded area surrounded by farm land. In November 1942, as part of World War II military activity, the US Navy established Camp Thomas which occupied 142 acres, including the Property, and was used as a troop staging area for Navy operations.

After World War II ended, the Property was not used until 1954 -1956 when the Army constructed a Nike missile battery. This Nike Battery Site PR-58 was equipped with short-range, conventionally-armed Nike Ajax missiles. The Nike Battery Site PR-58 operations included three underground missile magazines (Buildings 347, 348, and 349), a refueling area located to the south of Building 347, a missile assembly and test building (Building 344) with a 500 gallon #2 fuel oil underground storage tank (UST), a generator building with a 4,000 gal fuel oil UST (Building 345), and personnel quarters (Figure 4) (EA, 2001). In 1962, the Nike Ajax missiles were deactivated.

In 1964, the Property was transferred from the Army to the US Navy. The Navy's use of the Property was for Navy disaster recovery training which took place on the western side of the Property from 1964 until 1974. The Navy constructed five to six buildings for nuclear, chemical, and biological warfare training. Also, according to a report prepared by Sirrine Environmental Consultants (SEC, 1988), a small area (a 100 sq. foot concrete pad) of the Property was used for the storage of radioactive materials from 1968 to 1972 during the Navy's use of the Property.

In 1974, the land to the west of Seabee Avenue, including the Property, was transferred to the US General Services Agency (GSA). In 1978, the GSA transferred ownership of the Property to the Rhode Island Economic Development Corporation (RIEDC). The land remained unused by RIEDC until 1980.

Between 1980 and 1982, RIEDC leased 2.2 acres of the land to Peabody Clean Industries (PCI). PCI used the area as a hazardous waste storage and transfer facility (Figure 4). Available estimates indicate that, at any one time, a maximum of 50,000 gallons of oil/water waste, 10,000 gallons of waste solvents, waste oil and sludge,

and 20 drums of various other hazardous materials were stored on the Property. Figure 4 shows the three areas of PCI activity on the Property (Areas A-3, A-4 and A-5). Area A-3 contained four 20,000-gallon aboveground storage tanks (AST), designated "E", "F", "G", and "Z", which were used to store water, oil, and some solvent wastes. Area A-3 also contained an oil-water separator which was used for decanting the water phase from oily bilge water wastes to a three-trench leach field. Contaminated soil in two of the three trenches was removed, and the remaining one abandoned. Area A-4 included two buildings used by PCI to store non-hazardous equipment and supplies. The supplies were removed and the buildings demolished by the Rhode Island Port Authority (RIPA). Area A-5 comprised a concrete pad surrounded by a chain link fence used as temporary storage for drums of hazardous wastes (Environmental Resource Associates, Inc. (ERA), 1984).

In 1983, Rhode Island Department of Environmental Management (RIDEM) personnel directed storage tank closures and a cleanup of contaminated soil related to the PCI activities at the Property. These activities included the following:

- The four ASTs in Area A-3 were drained and the contents shipped to and disposed of at a licensed facility. The tanks were then cleaned and reportedly scrapped, except Tank "G", which was reportedly given to the State of Rhode Island;
- Ten cubic yards of contaminated soil from beneath the ASTs in Area A-3 were excavated and disposed of as hazardous waste, and the containment berms leveled;
- 710 cubic yards of soil from the leach field in Area A-3 were excavated and landfilled offsite The area around the AST, oil-water separator, and leach field was reportedly saturated to groundwater table with oils and water from the discharge. The excavation was terminated at the water table (Gates, 2010);
- The equipment stored in the buildings in Area A-4 was removed, and the buildings were demolished by RIPA;
- The drums stored in Area A-5 were removed and disposed of offsite, and the concrete pad and fence were demolished and removed (ERA, 1984).

The Property is currently not in use other than ongoing operations of a small commercial business located in the southwest corner of the Property.

2.3. Surficial and Bedrock Geology

The Property is relatively flat with slopes generally less than 1% and no major surface drainage features (although an extensive stormwater drainage system of swales, catch basins, and subsurface collection and conveyance piping that was constructed in the 1940s is still present today). There are scattered mounds and depressions across the Property, most of which are believed to be anthropogenic in origin. The three underground missile magazines still exist, although the Army sampled the contents of the magazines and removed hazardous materials, then demolished the concrete pads around the magazines, penetrated the bottom of the magazines, and backfilled them with sand and rubble in 1995. Therefore, there is currently little evidence of their presence at ground surface. The former missile magazine area is currently grass-covered and void of any structure. Other than a minimal pavement area associated with former buildings and the grassy area, some of the western portion of the Property is thickly covered with brush and small trees, and the remainder has been clear cut. Two areas on the western side of the Property have been regraded for the construction of drainage structures.

The boring logs from more than 200 locations, along with rock cores and limited outcrop data, were examined and used to evaluate the geology at and near the Property. A summary of this evaluation follows and a detailed description is presented in the CSM. The area is characterized by the unconsolidated deposits described above lying on the quartzitic and phyllitic bedrock of the Rhode Island Formation.

The unconsolidated deposits overlying bedrock were deposited during and shortly after the Wisconsin glacial stage, 10,000 to 12,000 years ago. The lowest stratigraphic unit is the glacial till which was deposited onto the scoured rock surface beneath the glacier. This till/weathered rock unit varies in thickness from less than a foot to more than 20 feet. The till is typically thicker on flat surfaces as compared with steeply dipping rock faces, and apparently has been eroded from portions of an on-Property northeast-southwest striking ravine in the bedrock surface.

The unconsolidated deposits include ice-proximal end moraine and esker gravel units which vary in thickness from less than a foot to more than 50 feet, and overlie the till. There are several sub-facies with lateral and vertical variation in grain size present. These ice-proximal deposits filled the on-Property bedrock ravine and also appear to extend close to the ground surface beneath the primary Source Area on the Property (the primary Source Area is defined in this document as the area inside the "inset box" on Figure 3a).

Above, and interfingering with, the ice-proximal gravels are fine-grained estuarine sediments which include a substantial component of silt and/or clay, but also often have very fine sand or fine sand as the primary constituent. These deposits are thickest to the east and thin or are absent to the north of the Property. The fine grained estuary deposits were not described as present beneath the Property primary Source Area ("inset" on Figure 3a).

The depth to bedrock ranges from ground surface at the outcrop locations, including one immediately southwest of the Property, to more than 70 ft below ground surface (fbgs). The Rhode Island Formation is characterized by cross-bedding and irregular, discontinuous beds. Observations from bedrock investigations show some beds are preferentially solution weathered and contain open holes, veins, and vugs. A number of fractures are present, both vertically oriented and tilted sub-parallel with the bedding and schistocity. Some fractures are open, or are partially filled with quartzite or calcite. Many of the fractures are extensive (greater than 30 feet long observed in outcrops), open (up to ⅛-inch wide) and partially filled with euhedral quartz crystals. Based on outcrop data, bedding has a wide range of strikes, and dips vary from moderate to steep, with most dips toward the east. Fracture orientation appears to comprise a conjugate set with northerly and easterly strikes, and steep dips; closest to the Property, dips are northwesterly, northerly, and easterly. Secondary porosity occurs along both bedding planes and fractures; secondary porosity density is greatest in the phyllite member of the formation.

2.4. Surface Water Hydrology

The Property lies within the Hunt River and Coastal drainage basins. All stream and river flow in this basin eventually discharges into Narragansett Bay.

There is currently no surface water on the Property. Historically, on-Property surface water flow and runoff was managed through stormwater control structures including extensive subsurface collection and conveyance piping. These structures remain in place, and were modified in 2011 to include a new retention basin in the southwest corner of the Property (Quonset Development Corporation (QDC), 2012a).

Surface water southeast of the Property comprises the Hall Creek drainage system, an interconnected series of modified natural ponds and streams which receives surface water runoff from the area via a system of

underground storm drains. The most recent Hall Creek drainage system modifications were made between 2010 and 2012, and the current conditions are depicted in Figure 5 (Quonset Development Corporation, 2012b). Available groundwater and surface water elevation data suggest that at some times, potentially-contaminated groundwater flowing southeast of the Property may discharge to the Hall Creek drainage system.

Two large wetlands are present northeast of the Property on the north side of Perimeter Road, and more wetlands are located farther to the north, which are downgradient of the PR-58 Property. Additional wetland areas are present to the west of the Property. Davol Pond is located 1,200 feet to the southeast on the Rhode Island Airport property and Allen Harbor is 3,000 feet to the east (see Figure 2).

2.5. Groundwater Hydrology

For the purposes of discussion and presentation, water level and water quality data have been divided into three hydrogeologic units:

1. Shallow overburden: "S"-series wells. Most of these wells are screened across the groundwater table, and almost all are screened above the silt/clay/sand unit.
2. Deep overburden: "D"-series wells. Most of these wells are screened at or within 5 feet of the bedrock surface.
3. Bedrock: "R and R2"-series wells. These wells are screened or have open holes within the bedrock.

On average, the depth to groundwater is greater than 6 feet, although depth to seasonal high groundwater in the spring in some areas can be less than 3 feet. Seasonal fluctuations of more than 8 feet have been noted in numerous wells within the Property while much less groundwater elevation variation is observed east and south of the Property.

Potentiometric maps of groundwater elevations in the shallow overburden, deep overburden, and bedrock aquifers were prepared using the most recent round of synoptic hydraulic data collected in 2010 (Figures 6a, 6b, and 6c). The potentiometric maps typically show hydraulic gradients from the northern boundary of the Property towards the southwest, south, southeast, and east in all three of the hydrogeologic units discussed above; a northeasterly flow component is also observed in the bedrock aquifer.

2.6. Conceptual Site Model

In developing the CSM, the observations regarding Property background and environmental setting each have implications for understanding the nature and extent of contamination and potential remedial strategies. The following text summarizes the CSM.

1. The primary source of contamination is 1,1,2,2 tetrachloroethane (TeCA) and trichloroethene (TCE) released in the vicinity of the Source Area to ground surface or shallow pits. Based on the presence of TeCA, which is not known to have been used at the Former Nike Battery PR-58 facility, but was a component of DANC (decontamination agent non-corrosive) likely used by the Navy, a release occurred between 1964 and 1974. Additional sources, both on the Property and off the Property, also may exist.
2. Based on multiple lines of evidence, residual dense non-aqueous phase liquids (DNAPL) containing TeCA and TCE may be present in the deep overburden and bedrock at the Source Area, and likely comprise the majority of the contaminant mass at the Property. The extent of

DNAPL is not defined; the extent and magnitude of chlorinated volatile organic compound (CVOC) concentrations in groundwater (e.g., EA-119D, MW03-14D, SES-03D, SES-09D) and soil (e.g., EA-102 and MW03-14), and rapid groundwater concentration rebound following pilot testing (e.g., EA-102D), DNAPL may have migrated beyond the currently uncertain limits of the Source Area (see Figure 7: Proposed Source Area Soil Boring Locations).

3. Property-related potential contaminants of concern (PCOCs) include TeCA, tetrachloroethene (PCE), TCE, and their degradation products in soil gas, groundwater, surface water, and sediments. The only PCOC metal identified in Property soils was cobalt, which was detected at only one sampling location and at concentrations less than its ecological risk screening level (RSL). Worksheets #15-A through 15-Y of the QAPP include the Project Action Limits for each PCOC, as well as their human health and ecological RSLs, with citations of the source of each RSL.
4. Although the bottom of the CVOC contamination has not been documented at the Source Area, extrapolation based on uncontaminated downgradient wells combined with detections in the deepest wells in the Source Area indicates that the maximum depth of CVOC contamination is at least 105 fbgs, equivalent to approximately -75 feet NAVD (North American Vertical Datum) elevation. No further investigations were made through the Source Area in order to avoid potentially allowing further downward migration caused by drilling and well installations.
5. The lateral extent of the CVOC plume in groundwater and surface water has been well defined, with non-detections or detections below laboratory practical quantitation limits and/or maximum contaminant levels (MCLs) documented in all directions (see Figure 8: Contaminant Plume Maps). However, based on the absence of shallow groundwater monitoring wells on the NCBC property within approximately 1,000 feet east of the Property, and on reported historical activities at NCBC Sites, uncertainty exists regarding the adequacy of the off-Property shallow groundwater monitoring network in this area.
6. The extent of dissolved metals in deeper groundwater has been defined. Because sampling procedures used historically to collect shallow groundwater samples for dissolved metals analysis appear to have resulted in entrainment of solids in the groundwater samples, reported metals concentrations in shallow groundwater samples are not considered representative of shallow groundwater quality at the Property.
7. No MCLs were exceeded in drinking water samples from private wells tested in 2009. Only one contaminant was detected in samples from those wells: chloroform in the sample from well WP-9 at a concentration an order of magnitude below the MCL for total trihalomethanes. Because the well is located upgradient of the Source Area, the low concentration of chloroform in the water sample is not considered related to the Property. It is noted that there are several private wells in use whose owners did not allow access for sampling in 2009. Additionally, the private well samples that were collected were whole-well samples (rather than discrete interval samples) and the sample locations were determined by the existing well locations rather than necessarily at the optimum locations for monitoring groundwater quality.
8. MCLs for TCE, cis-1,2-dichloroethene (DCE), trans-1,2-DCE, 1,1-DCE, 1,1,2-trichloroethane (TCA), and vinyl chloride (VC) were exceeded in the most recently sampled groundwater on the

Property and/or within the RIDEM GB Groundwater area. RIDEM Class GB upper contaminant levels (UCLs) for TCE were also exceeded.

9. MCLs for TeCA, PCE, TCE, cis-1,2-DCE, trans-1,2-DCE, 1,1-DCE, 1,1,2-TCA, and VC were exceeded in the most recently sampled groundwater in the Property vicinity and/or within the RIDEM GB Groundwater area. RIDEM Class GB UCLs for TCE were also exceeded in groundwater on the Property and its vicinity.
10. MCLs for TCE, and possibly cis-1,2-DCE (based on historical data) were exceeded in the RIDEM GA-classified aquifer below the undeveloped portion of the Carriage Hill Association (CHA) common land immediately north of the Property. Access was not provided between 2008 and 2010 onto the CHA common land to confirm current groundwater quality with respect to MCLs. MCLs were not exceeded in the well cluster located in the Candlewood Drive cul-de-sac, at the residential area further northeast of the Property. The CHA common land has land restrictions that require the approval of the Town prior to development (CHA, 1981).
11. Groundwater divides and apparent limits of DNAPL migration extent have apparently protected groundwater beneath the neighborhoods to the northwest, west, and southwest of the Source Area from contamination exceeding MCLs.
12. Dissolved CVOC contaminants migrate in groundwater from the DNAPL in the Source Area to the northeast, east, and southeast, largely following bedrock hydraulic gradient directions.
13. The bedrock and deep overburden aquifers are hydraulically connected. Contaminant mass migration appears to primarily occur in the deep overburden and bedrock; however, upward vertical hydraulic gradients (measured in October 2009 and shortly after record precipitation in April 2010) and groundwater discharge to natural discharge areas at wetlands and surface waters downgradient of the Property result in CVOC migration from the Property to shallow groundwater, pore water, and surface water in the vicinity of Hall Creek and Davol Pond. It is possible that similar discharges occur to wetlands on the CHA common land northeast of the Property, but these could not be assessed because access to this area was denied by the property owners.
14. Surface water guidelines were not exceeded by Property-related CVOC PCOCs. Sediment quality guidelines were marginally exceeded at only one of six locations, in Hall Creek, by TCE (97.3 micrograms per Kilogram (µg/Kg) reported, compared with the 96.9 µg/Kg sediment quality guideline).
15. CVOCs have been detected in sub-slab soil gas samples collected south of the Property, beneath the North Kingstown Department of Public works (DPW) building. Volatilization of CVOCs from dissolved contamination at the groundwater table is one possible source of CVOCs in these sub-slab soil vapor samples. The available groundwater analytical results are insufficient to characterize the extent of volatile organic compounds (VOC) concentrations in groundwater at the North Kingstown DPW facility. Available data indicate that Property-related shallow groundwater CVOC contamination is not present under the residences located southwest, west, northwest, north, and northeast of the Property, and that soil vapor intrusion risks in residential neighborhoods are limited or non-existent.
16. Order-of-magnitude estimated travel times of 20 to 40 years for the leading edge of the CVOC plume in overburden groundwater match the likely time period of release at the Source Area.

17. Available time series data indicate the presence of a mature plume with abundant degradation products and declining concentrations in most areas. However, the data also suggest that contamination may have been remobilized in the bedrock by the 2005 Steam Enhanced Recovery (SER) pilot test, and that several wells, particularly overburden wells within the Source Area and wells to the east-southeast and southeast of the Source Area, had higher CVOC concentrations in 2009 than in 2007. Hence, this portion of the plume may not be in a state of dynamic equilibrium and further changes in its magnitude and extent could be expected.
18. Previous investigations of the NCBC Sites 01, 02, 03, 04, and 16 (EA, 1998; TetraTech-NUS, 2008) have provided an appropriate metals background data set for groundwater, soil, surface water, pore water, and sediment for the two soil types mapped on the Property.

3. DATA GAP EVALUATION

Despite the challenges stemming from the limited information regarding the historical uses of the Property, which dates back through more than 65 years of Army and Navy use, the available information regarding historical activities at the Property (Nike operations, Navy disaster training, and Peabody Cleaning, Inc. hazardous waste storage), as well as results from prior investigations, suggest multiple releases of a variety of products/compounds (e.g., TCE, DANC, etc.) over many years. Based on these data, the Source Area has been generally defined during prior investigations, as comprising an area of significantly higher concentrations of contaminants in soil, rock, and groundwater. The three activities described above were generally located in the same portion of the Property. Based upon currently available information, the 2011 CSM identifies one "Source Area" (depicted in Figures 2 and 7).

3.1. Data Gaps Identified in the *Draft RI Report*

Review of the *Draft RI Report* (JCO, 2011) and associated CSM indicates that a number of data gaps remain for the Project. These data gaps include the following:

1. According to Subsection 3.1.30 of the *Draft RI Report*, interviews with personnel associated with the soil boring at location EA-102D indicate that a zone of very high concentrations and potentially non-aqueous phase liquids (NAPL) is present in the 10 to 14-ft depth interval in the vicinity of the concrete pad. This data gap is identified in Task 2 of the *PWS*, and the investigatory activities, specifically, a subsurface investigation in the vicinity of soil boring EA-102D, are provided for under Task 7 of the *PWS*.
2. As noted in item 6 of Section 2.6 above, sampling procedures used historically to collect shallow groundwater samples for dissolved metals analysis appear to have resulted in entrainment of solids in the groundwater samples. Reported dissolved metals concentrations in shallow groundwater samples are therefore not considered representative of shallow groundwater quality at the Site. This data gap is identified in Task 2 of the *PWS*, and the investigatory activities, specifically, collection of a comprehensive set of low-turbidity shallow groundwater samples, are provided for under Task 7 of the *PWS*.
3. As noted in item 15 of Section 2.6 above, USACE has determined that available groundwater analytical results are insufficient to characterize the extent of volatile organic compounds (VOC) concentrations in groundwater at the North Kingstown DPW facility. This data gap is identified in Task 2 of the *PWS*, and the investigatory activities are provided for under Task 16 of the *PWS*, as follows: Stone shall install, develop, survey, and sample for VOCs, two (2) shallow monitoring wells (depths not to exceed 30 fbgs) at the North Kingstown DPW facility located on Babcock Road, in North Kingstown, RI.
4. The bottom of the CVOC contamination has not been documented at the Source Area. No further investigations were made through the known Source Area in order to avoid potentially allowing further downward migration caused by drilling and well installations.
5. In Subsection 5.3.2 of the *Draft RI Report* (JCO, 2011), the Screening Level Ecological Risk Assessment (SLERA) included a recommendation that additional samples for CVOC analyses of pore water, surface water, and sediment be collected from Hall Creek during "worst case" seasonal low groundwater conditions to confirm the apparent minimal impact to ecological receptors. Additionally, the SLERA recommended measurements of the vertical hydraulic

gradient be made at the time of sample collection. If CVOC concentrations under seasonal low groundwater conditions are found to be above ecological screening levels, a baseline ecological risk assessment would need to be performed to further evaluate the potential for risks to ecological receptors.

3.2. Data Gaps Identified in the *Data Gap Analysis Report*

Stone's *Draft Data Gaps Analysis Report* (Stone, 2012) identified twelve remaining data gaps for the Property. The data gaps include the following:

1. Dissolved metals in shallow aquifer: As noted in item 6 of Section 2.6 above, samples collected to assess the concentrations of metals in shallow groundwater were obtained using bailers. This resulted in samples with relatively high turbidity, which in turn resulted in elevated concentrations for some inorganic compounds above MCLs. Since analysis of turbid samples result in data that are not representative of true dissolved metals concentrations, the data available are not reliable for assessing the concentrations of metals in shallow groundwater. Shallow monitoring wells need to be sampled using the USEPA Low Stress/Low Flow method, which is designed to collect representative groundwater samples that reflect ambient flow conditions with minimal physical and chemical alterations caused by sampling, particularly reduced turbidity, in order to properly assess metals concentrations in shallow groundwater. This data gap is identified in Task 2 of the *PWS*, and the investigatory activities, specifically, collection of a comprehensive set of more representative, low-turbidity shallow groundwater samples, are provided for under Task 7 of the *PWS*.
2. Hall Creek Ecological Risk: In Subsection 5.3.2 of the *Draft RI Report* (JCO, 2011), the SLERA included a recommendation that additional samples for CVOC analyses of pore water, surface water, and sediment be collected from Hall Creek during "worst case" seasonal low groundwater conditions to confirm that contaminants of concern (COCs) present only a minimal impact to ecological receptors. Additionally, measurements of the vertical hydraulic gradient should be made at the time of sample collection. If CVOC concentrations under seasonal low groundwater conditions are found to be above ecological screening levels, a quantitative baseline ecological risk assessment would need to be performed to further evaluate the potential for risks to ecological receptors. This data gap is identified in Task 2 of the *PWS*, and the investigatory activities are provided for under Task 7 of the *PWS*, as follows: Stone shall collect co-located pore water, surface water, and sediment samples at the 12 locations previously sampled by JCO, but at a time period that meets the following criteria: no significant precipitation for at least 3 days; lower than mean stream stage; and, falling groundwater elevations.
3. Insufficient Source Area Characterization: According to Subsection 3.1.30 of the *Draft RI Report*, interviews with personnel associated with the soil boring at location EA-102D indicated that a zone of very high concentrations and potentially non-aqueous phase liquids (NAPL) is present in the 10 to 14-ft depth interval in the vicinity of the concrete pad. Personnel from the well drilling crew that installed monitoring well EA-102D informed USACE that they had observed NAPL in the settling tub while advancing casing between 10 and 14 ft bgs. Monitoring well EA-102D is located near the northeast portion of the former concrete pad (see Figure 7). No samples were collected from the 10 to 14 foot interval during drilling of the well. The screened interval for EA-102D spans approximately 22 fbgs to 32 fbgs (i.e., below the zone of reported

NAPL). Therefore, hard data are not available with which to determine whether or not such NAPL is present and if so what the composition of the NAPL is. The proposed Source Area NAPL characterization will be undertaken to verify the presence or absence of NAPL in the vicinity of EA-102D, and, if present, to identify the physical and chemical properties of NAPL and delineate its horizontal and vertical distribution. This data gap is identified in Task 2 of the *PWS,* and the investigatory activities — specifically, a subsurface investigation in the vicinity of soil boring EA-102D — are provided for under Task 7 of the *PWS*. The possibility that NAPL was drawn vertically downward during drilling will be evaluated, during the investigation, by advancing the initial Source Area soil boing as close as possible well EA-102D, with careful NAPL screening performed along the entire length of the boring.

In addition, the geometry of the Source Area is not well defined to the north and east of monitoring well EA-119D (see Figure 7), limited soil VOC data are available within the "core" of the Source Area to evaluate contaminant mass distribution. These additional data would be useful in evaluating Source-Area remedial alternatives during the FS, and will be obtained during the RI/FS field program.

4. Lack of Soil Treatment Parameters in the Source Area: There are insufficient data to evaluate the feasibility of certain remedial alternatives which may be assessed for use in the Source Area. Certain data, such as bedrock fracture interconnectivity data, while important to evaluation, but more so to the design of certain possible remedies, are expensive to obtain and should be deferred until such a remedy passes other screening steps in the FS process. However, other data, such as soil oxidant demand, are relatively inexpensive to obtain, particularly if combined with the collection of other data in the Source Area.
5. Minimal Water Quality Data from Certain Wells: Approximately 250 wells have been installed on and around the Property over the course of the environmental investigations. However, some of these wells have a limited sampling history. For example, the wells in the CHA wetlands have only been sampled twice, due to inability to obtain access from the property owner, and the cluster of wells along Candlewood Drive has only been sampled once. With such a limited data set it is not possible to assess trends in contaminant concentrations. More current data are needed to determine these trends and current potential for risk, as well as to assess the presence or absence of contaminants. A round of sampling of selected wells, including those with sparse sampling histories, is needed.
6. Current Biological / Geochemical Data to Evaluate Natural Attenuation: Three previous assessments of the nature and role of biodegradation in attenuation of groundwater contamination associated with releases at the PR-58 Property have indicated that biodegradation is occurring and may potentially be a significant mechanism for limiting groundwater transport of contaminants. The first two of these assessments were conducted 14 and 12 years ago, respectively. The most recent assessment was based only on the presence of degradation products and included data collected between 1996 and 2010. There is a need for current biological and geochemical data accompanying current data regarding contaminant concentrations to allow for an assessment of the degree of biodegradation and the likelihood of degradation acting as a significant means of plume attenuation.

7. Lack of Primary Porosity Data from the Bedrock with which to Assess the Potential for Matrix Diffusion: It is likely that DNAPL entered fractures in the bedrock during the release history at the PR-58 Property. The bedrock at the PR-58 Property is described as dark grey-black, contorted phyllites/silty sand turbidites (greywacke) and dark grey-black, carbonaceous laminated phyllites and meta-argillites with pyrite and graphite. Bedrock core logs, outcrops, and borehole optical and acoustical televiewer logs show some beds are preferentially solution-weathered and contain open holes, veins, and vugs. These rocks may contain primary (matrix) porosity on the order of 1% to 10%. This degree of porosity is sufficient to allow for diffusion of a large amount of contaminant mass from the dissolution of the DNAPL in the fractures into the pore spaces in the matrix. If present in the matrix porosity, this contaminant mass represents a long-term source of contamination to groundwater in the bedrock fractures. Additionally, it would be very difficult, if not impractical, to remediate this contaminant mass. No data regarding the porosity of the rock matrix exist for the PR-58 Property. These data can be generated from existing rock core from the Property that USACE has stored at Fort Devens, Massachusetts. Measurements of porosity of selected portions of the rock core should be made to allow for a determination of the potential for matrix diffusion to play a significant role in source persistence and the likely performance of source area remedial measures.
8. Groundwater/Plume Discharge to CHA Wetlands: Current groundwater conditions on CHA property are unknown due to refusal by the CHA to grant access for purposes of data collection on their property. Existing data indicate that groundwater from the bedrock likely discharges upward into the deep unconsolidated deposits on the CHA property. It is unknown whether groundwater in the deep overburden flows upward into the shallow overburden and subsequently discharges into the wetlands on the CHA property. If such a condition exists, it would present a potential exposure pathway. However, monitoring wells on the CHA property have not been sampled since November 2007, due to the refusal of CHA to grant access for sampling. Thus, current plume conditions north of the PR-58 Property are unknown. Both hydraulic and water quality data are needed at the wetlands and other areas on the CHA property to assess this potential.

   However, AECOM's 2011 ecological risk assessment states "The wetlands located north of Perimeter Road on the Carriage Hill Association common land could not be sampled due to Right of Entry issues. Therefore, a quantitative evaluation of wetland-specific data could not be conducted. Due to the lack of wetland-specific analytical data, there are some uncertainties in this evaluation. CVOCs were not detected in surface water collected from northeast of the Site in 2007 and the TCE plume at the north of the Site is located in the deep overburden and is therefore unlikely to discharge into the wetlands. Based on this information, it does not appear that these wetlands would be adversely impacted by Project-related CVOCs in groundwater. Therefore, ecological evaluation is not warranted for these wetlands." (AECOM, 2011). The combination of lack of access to suitable sampling locations on CHA property, 2007 data indicating no impacts to these wetlands, and AECOM's conclusions eliminate further consideration of this data gap.
9. Shallow Groundwater Quality at the North Kingstown DPW Property: In April 2010, USACE undertook sub-slab soil gas sampling beneath the DPW building. Four COCs related to the PR-58 Property were detected above Risk-Based Concentrations (RBCs) for sub-slab soil gas. These

four compounds are TeCA, chloroform, PCE, and TCE. Based on data from monitoring wells OB-ED and MW-DPT-2, the contaminant plume from the PR-58 Property flows through the deep unconsolidated deposits and the bedrock under the DPW property. As noted in item 15 of Section 2.6 of this Work Plan, USACE has determined that available groundwater analytical results are insufficient to characterize the extent of CVOCs concentrations in groundwater at the North Kingstown DPW facility. Shallow groundwater data are needed to determine if a groundwater-soil gas-indoor air pathway exists for PR-58 contaminants at the DPW property. This data gap is identified in Task 2 of the *PWS*, and the investigatory activities are provided for under Tasks 16 and 27 of the *PWS*, as follows: Stone will advance soil borings, collect and analyze one soil sample from each boring for VOCs, and install, develop, survey, and sample for VOCs, two (2) shallow monitoring wells (depths not to exceed 30 fbgs) at the North Kingstown DPW facility located on Babcock Road, in North Kingstown, RI.

10. Incomplete Human Health Risk Assessment for the PR-58 Property and Areas East and South/Southeast of the PR-58 Property: The PR-58 Property has been subdivided, and foreseeable future uses have become better defined. To accommodate these developments, as well as to incorporate the new analytical data being collected as part of this project, the existing Human Health Risk Assessment (HHRA) for the PR-58 Property, completed by AECOM in 2011, will be updated. Three potential exposure areas are identified on the PR-58 Property: the Recreational Area, the Rest of Nike Area, and the Source Area. The following components of a HHRA must be completed to understand risks to human health in these areas:

    a. The three potential exposure areas evaluated for HHRA on the PR-58 Property require an evaluation of the following exposure scenarios:

        i. indoor worker;

        ii. construction worker; and

        iii. child through teenager trespasser.

    b. In addition, the Recreational Area and any portion of the Source Area that extends onto the parcel designated for development as ballfields require an evaluation of health risks posed to children and teenagers using the area for recreation.

    c. If wells are to be installed on the PR-58 Property for purposes of irrigation of the ballfields, the irrigation water pathway must be assessed for risks to children and teenagers using it for recreation.

    No further environmental data will be required to evaluate potential soil vapor migration impacts, and the potential risks posed by soil vapor migration in this area will be performed as part of a revised HHRA, which will be included with the Final RI Report.

11. The groundwater plume originating at the Source Area at the PR-58 Property migrates to the east, south, and southeast. Properties in these areas may undergo further development. An assessment of human health risks due to soil exposure is being completed by contractors to the Navy (TetraTech, 2012). However, an assessment of human health risks due to potential soil vapor migration from the groundwater plume that underlies these areas has not been completed. No further environmental data will be required to evaluate potential soil vapor migration impacts,

and the potential risks posed by soil vapor migration in this area will be performed as part of a revised HHRA, which will be included with the *Final RI Report*.

12. Permission Denied to Monitor Several Water Supply Wells: The Rhode Island Department of Health (RIDOH) sampled eight private water supply wells for VOCs in 1997. The five wells located closest to the PR-58 Property had no detections of VOCs, nor did the two northeastern-most private wells along Fletcher Road. Trace concentrations of PCE (0.7 µg/L) and 1,1,1-TCA (1 µg/L) (1,1,1-TCA is not a PR-58-related contaminant) were measured in the sample collected from a well (WS-4) located more than 2,000 feet north-northeast of the Property at 550 Fletcher Road. In 2006, USACE requested permission to resample the eight wells and was granted permission to sample only three. The three private water supply wells (WS-6, WS-9, and WS-18) for which access for sampling was granted were sampled in September 2006. The wells were located at residences along Fletcher Road. No CVOCs were detected in the samples from WS-6 and WS-18. The only VOC detected in the sample from WS-9 was chloromethane (not a PR-58-related contaminant). In 2009, access was granted to sample seven private water supply wells: three wells located on Fletcher Road that had been sampled previously in 1997 and 2006 (WS-6, WS-9, and WS-18), and four new wells, including three located on Potter Road. However, permission has not granted to sample WS-4 since 2006 so conditions at this and other locations where access has been denied have not been updated.
13. Lack of Data on the Vapor Intrusion Exposure Pathway on Areas North of the PR-58 Property (CHA Land): The refusal of the CHA to grant access to properties north of the PR-58 Property has resulted in data gaps regarding the groundwater plume in that area. Current concentrations of COCs in the deep unconsolidated deposits and the bedrock are unknown. In addition, the vertical component of the hydraulic gradient between deep unconsolidated deposits and the shallow unconsolidated deposits is unknown. If these gradients are upward, the plume may rise into the shallow unconsolidated deposits and reach a position at or near the water table. Under such conditions, volatilization of the contaminants from the water table/capillary fringe into the soil gas could occur. Soil gas contamination near residences could enter the buildings either through basements or slabs and cause a degradation of indoor air quality, posing a risk to residents. This pathway cannot be adequately assessed without data on shallow groundwater quality, hydraulic gradients, and soil gas concentrations. Groundwater quality and hydraulic gradient data would be collected first to ascertain whether a near-water-table plume exists in the area. If so, soil gas data would need to be collected as the next step in determining if a potential exposure pathway is present.
14. Limited Shallow Monitoring Well Network East of PR-58 Property: The area east of the PR-58 Property was operated by the NCBC and experienced activities that may have resulted in releases of contaminants. Regarding transport east from the PR-58 property, the *Draft RI Report* (JCO, 2011) concluded:
    a. Transport east beneath NCBC Sites 02 and 03 is primarily due to flow through the bedrock.
    b. Migration beneath NCBC Sites 02 and 03 in the deep unconsolidated deposits is primarily through interconnected sandy gravel (and/or sand) units, which may be discontinuous.
    c. There is a separate source of CVOCs (including TeCA) in the vicinity of NCBC Sites 03 and/or 02.

Thus, the actual migration pathways and extent of contamination originating at the PR-58 Property is incompletely understood. In addition, there is a high likelihood of additional sources of contamination on NCBC Sites 02 and 03. The existing groundwater monitoring network east of the Property is not sufficient to resolve these issues.

4. REMEDIAL INVESTIGATION / FEASIBILITY STUDY FIELD PROGRAM OBJECTIVES AND TASK SUMMARY

4.1. Objectives of the Remedial Investigation / Feasibility Study Field Program

As previously described in subsection 1.1, the objectives of the RI/FS field program are to:

1. Address data gaps raised in the *Draft Data Gap Analysis Report*, dated May 25, 2012;
2. Collect data necessary to support an FS, Proposed Plan, and Decision Document, as outlined in the *Performance Work Statement (PWS)*, issued by USACE on November 5, 2010 and subsequently modified on October 31, 2011, December 12, 2011, March 26, 2012, June 15, 2012, and July 11, 2012.

4.2. Remedial Investigation / Feasibility Study Field Task Development

The tasks described in the Work Plan comprise the list of investigative tasks that are required to achieve the objectives of the RI and FS, namely, to eliminate the identified data gaps and to identify and narrow the list of potential remedial technologies. The list of field tasks has been developed in an iterative fashion, with input and approval from USACE and Stone, through the following steps:

1. Initial SOW tasks were included in the *PWS*, dated November 5, 2010;
2. Subsequent discussions between USACE and Stone resulted in revisions to the *PWS*, dated on October 31, 2011, December 12, 2011, March 26, 2012, June 15, 2012, July 11, 2012, January 29, 2013, and June 19, 2013;
3. Additional data gaps were identified by Stone under Task 2 of the *PWS*, summarized in the *Draft Data Gaps Analysis Report*, dated May 25, 2012 (Stone, 2012);
4. In a conference call with Stone on August 16, 2012, USACE requested that, in addition to evaluating the possible presence of NAPL in the vicinity of monitoring well EA-102D, the purpose of the "Additional Source-Area Characterization" task will be to further refine the geometry of the Source Area (primarily to the north and east). Further refinement of the Source-Area geometry will be funded via Optional Task 19 of Mod 01 ("Expanded Source-Area Investigation").
5. In a conference call with Stone on November 7, 2012, USACE requested that, in addition to collecting co-located pore water, surface water, and sediment from the vicinity of Hall Creek, an additional set of samples be collected from a wetland to the north of the Property via private property at 710 Fletcher Road, to be funded via Task 7 of Mod 01.

4.3. Remedial Investigation / Feasibility Study Field Task Summary

Using the above process, the RI/FS field tasks were developed to address the data gaps identified in Section 3. Many of the tasks serve to eliminate more than one data gap, and many also provide further benefits in addition to data gap reduction. It should be noted that data gap 12 is being partly addressed as part of the RI/FS investigation due to property access issues. Data gap 10 is being addressed through a separately-scoped update to the HHRA. The following list summarizes the seven RI/FS field tasks:

1. Collection and analysis of additional pore water, surface water, and sediment samples from Hall Creek and vicinity;

2. Collection and analysis of pore water, surface water, and sediment samples from a wetland north of the Property;
3. Analysis of existing rock core for primary porosity;
4. Collection and analysis of groundwater samples from approximately 50 monitoring wells;
5. Collection and analysis of water samples from residential supply wells;
6. Additional Source Area characterization;
7. Shallow groundwater assessment at the North Kingstown DPW facility;
8. Synchronous groundwater level measurements from approximately 50 monitoring wells (in addition to the 50 wells to be sampled (Item 4); and
9. Description and measurement of a newly-exposed bedrock outcrop west of the Source Area.

Table 1 identifies the data gaps that need to be addressed, the objective of the data gap reduction, additional benefits of the action, and the specific task(s) employed to address the data gap. Data quality objectives (DQOs), which further detail the objectives for each field task, are described in Worksheet # 16 of the QAPP.

Table 1: Data Gaps and Collection/Assessment Tasks and Methods

Data Gap	Objectives	Additional Purpose(s)	Data Collection Task and Methods
1) Questionable representativeness of dissolved metals results from the shallow aquifer.	Confirm dissolved metals concentrations in shallow groundwater.	Provide additional groundwater quality data for use in revised HHRA.	Tasks: Monitoring well sampling and analyses. Methods: Low-flow groundwater sampling, water level measurement, sample location, sample analysis
2) Lack of "worst case" pore water, surface water, and sediment quality data from Hall Creek.	Obtain pore water, surface water, and sediment quality data and vertical hydraulic gradient data that will be used to evaluate risk to ecology of creek during "worst case" seasonal low groundwater conditions.	Provide additional understanding of contaminant transport pathways.	Tasks: Pore water, surface water, and sediment sampling and analyses with hydrological evaluation of hydraulic gradients. Methods: Ekman sampler for sediments, grab sampling for surface water and Henry sampler for pore water sample collection. All samples analyzed for VOCs, sediment samples analyzed for TOC, and surface water samples analyzed for hardness. Mini-piezometers will be used for the evaluation of hydraulic gradients in the creek bed.
3) Insufficient Source Area Characterization.	Determine extent of NAPL and source zone contamination in the vicinity of monitoring well EA-102D; if NAPL is present, analyze NAPL for a broader suite of composition and physical properties. Better define extent of Source Area north and east of monitoring well EA-119D. Improve understanding of VOC mass distribution within Source.	Provide source area treatability data.	Tasks: Soil coring and analyses, monitoring well installations, monitoring well sampling. Methods: Soil coring and sampling with VOC analyses. Oil red-O dye staining for NAPL identification. If present, NAPL collection and analyses for a broader suite of chemical composition and physical properties. Subset of soil samples also analyzed for TPH, physical properties, and TOC.
4) Lack of Soil Treatment Parameters in the Source Area	Obtain soil treatability data in the Source Area.		Tasks: Soil coring and analyses Methods: Soil coring and sampling with soil oxidant demand analyses.
5) Insufficient groundwater quality data from certain areas/wells.	Provide additional groundwater quality data for use in revised HHRA.	Provide additional groundwater quality data for use in revised HHRA.	Tasks: Monitoring well sampling and analyses. Methods: Low-flow groundwater sampling with VOC analyses at select monitoring wells.
6) Lack of current geochemical data to evaluate natural attenuation.	Obtain current geochemical data to update the evaluation of whether natural attenuation is playing a significant role for limiting plume migration.	Evaluate potential groundwater and vapor intrusion contamination. Provide additional understanding of contaminant transport pathways.	Tasks: Monitoring well sampling and analyses. Methods: Low-flow groundwater sampling with VOC and biodegradation parameters (*Dehalcoccoides* (qDHC), TCE reductase (tceA), vinyl chloride reductase (bvcA), and vinyl chloride reductase (vcrA) analyses. Field analyses of nitrate/nitrite, sulfate/sulfite, ferrous/ferric iron, and chloride measurements.

Data Gap	Objectives	Additional Purpose(s)	Data Collection Task and Methods
7) Lack of primary porosity data from the bedrock.	Obtain porosity data to assess potential for matrix diffusion.	Provide additional understanding of contaminant transport pathways. Provide source area treatability data.	Tasks: Sampling and analyses of existing rock cores. Methods: Rock core sampling, sample analysis.
8) Groundwater/Plume Discharge to CHA Wetlands	Obtain pore water, surface water, and sediment quality data and vertical hydraulic gradient data that will be used to evaluate risk to human health and the environment.	Provide additional understanding of contaminant transport pathways.	Tasks: Pore water, surface water, and sediment sampling and analyses; hydrological evaluation of hydraulic gradients. Methods: Ekman sampler for sediments, grab sampling for surface water and Henry sampler for pore water sample collection. All samples analyzed for VOCs, sediment samples analyzed for TOC, and surface water samples analyzed for hardness. Mini-piezometers will be used for the evaluation of hydraulic gradients in the wetland.
9) Lack of shallow groundwater quality data at the DPW property	Obtain shallow groundwater quality data which are needed to determine if vapor intrusion pathway for Property-related contaminants exists at the DPW property.	Provide additional groundwater quality data for use in revised HHRA. Provide additional understanding of contaminant transport pathways.	Tasks: Installation and sampling of two new shallow overburden monitoring wells at the DPW property. Methods: Slow purge groundwater sampling with VOC analyses
11) Lack of groundwater quality data for several residential water supply wells.	Obtain groundwater quality data at several residential supply wells where access has previously been denied.	Provide additional groundwater quality data for use in revised HHRA. Provide additional understanding of contaminant transport pathways.	Tasks: Residential water supply well sampling. Methods: Groundwater sampling from residential supply wells with VOC analyses.
12) Lack of “worst case” pore water, surface water, and sediment quality data from northern wetlands.	Obtain pore water, surface water, and sediment quality data and vertical hydraulic gradient data that will be used to evaluate risk to ecology of wetlands during “worst case” seasonal low groundwater conditions.	Provide additional understanding of contaminant transport pathways.	Tasks: Pore water, surface water, and sediment sampling and analyses with hydrological evaluation of hydraulic gradients. Methods: Ekman sampler for sediments, grab sampling for surface water and Henry sampler for pore water sample collection. All samples analyzed for VOCs, sediment samples analyzed for TOC, and surface water samples analyzed for hardness. Mini-piezometers will be used for the evaluation of hydraulic gradients in the wetland.
13) Limited Shallow Monitoring Well Network East of PR-58 Property	Improve the monitoring well network at NCBC Sites 2 and 3	Provide additional understanding of contaminant transport pathways	Communicate perceived data gaps to US Navy Project Managers, so that they can be addressed by US Navy investigations.

5. REMEDIAL INVETIGATION / FEASIBILITY STUDY FIELD TASK DESCRIPTIONS

The RI/FS field tasks described in this section are designed to address the data gaps identified in Section 3 and summarized in Table 1. Certain aspects of this investigation will follow the Triad Approach, i.e., some of the data collection tasks will be determined based on the results of previous analytical results from this investigation. This interdependency is indicated in Table 1. In the interest of reducing redundancy, the following subsections contain a summary of the RI/FS field tasks to be performed; the SAP (Appendix A) contains the details regarding how each field task of the investigation will be performed and will be the primary reference for field personnel. A summary of the overall sequencing of the primary work elements is as follows:

1. Collection and analysis of additional pore water, surface water, and sediment samples from Hall Creek and vicinity;
2. Collection and analysis of additional pore water, surface water, and sediment samples from a wetland north of the Property;
3. Analysis of existing rock core for primary porosity;
4. Collection and analysis of groundwater samples from approximately 50 monitoring wells;
5. Collection and analysis of drinking water samples from residential supply wells
6. Additional Source Area characterization; and
7. Shallow groundwater assessment at the North Kingstown DPW facility;
8. Collection of synchronous groundwater elevation data from approximately 50 wells (in addition to those sampled under Item 4); and
9. Description and measurement of a bedrockoutcrop recently exposed west of the Source Area.

Consistent with the Triad Approach, many of the field tasks will require decisions that are dependent on the results of prior tasks. For instance, during subsurface soil investigations to determine the nature and extent of NAPL in the vicinity of soil boring location EA-102D, the location of each subsequent soil boring will be based upon the near-real-time results of the previous soil borings, and the decision to terminate the program will be made based on the same data, in this case, with approval of USACE. Every effort will be made to keep RIDEM and EPA informed of progress. Data will be provided electronically, along with preliminary interpretations and recommendations. Telephone conferences will be utilized when there are differences of opinion as to the preferred next step in the investigation. However, these decisions will be of a “time critical” nature due to the presence of multiple Stone personnel, equipment and subcontractors onsite. Therefore, if timely responses are not provided by all stakeholders, the work will proceed upon the agreement of those stakeholders available.

5.1. Hall Creek Investigation

Twelve co-located pore water, surface water, and sediment samples will be collected along Hall Creek, depicted on Figure 5. The proposed locations are the same locations previously sampled by JCO in 2009. The samples will be collected no earlier than June, during the seasonal low-flow period for surface water, which will be determined by monitoring precipitation, surface water stage, and groundwater elevation data in the vicinity of the Property, and which will be confirmed with CENAE before sampling commences. Stone will

collect a hydrological dataset at the time of sampling that will document the achievement of DQOs for this sampling task. The sediment samples will be collected from the "bioactive" zone, 15 to 20 cm below the sediment-water interface. All the samples will be analyzed for CVOCs via USEPA Method 8260B by the fixed laboratory; in addition, six of the pore water samples will be analyzed for Target Analyte List (TAL) metals by USEPA Methods 6010/7470/7000, the surface water samples will be analyzed for hardness via USEPA Method 6010, and the sediment samples will be analyzed for total organic carbon via the Lloyd Kahn Method. Information regarding the laboratory requirements for detection limits, precision, and accuracy of pore water samples are summarized in Table 5, found in Subsection 2.10 of the SAP, and discussed in Subsection 2.10 of the SAP and the QAPP (Appendix A).

5.2. Northern Wetland Investigation

A co-located set of pore water, surface water, and sediment samples will be collected from a wetland located north of the Property, east of Fletcher Road, depicted on Figure 5. The proposed locations are the same locations previously sampled by JCO in 2009. The samples will be collected synchronously with, and in the same manner as, the Hall Creek investigation, if possible. The samples will be analyzed for CVOCs via USEPA Method 8260B by the fixed laboratory; in addition, the pore water sample will be analyzed for TAL metals by USEPA Methods 6010/7470/7000, the surface water samples will be analyzed for hardness via USEPA Method 6010, and the sediment samples will be analyzed for total organic carbon via the Lloyd Kahn Method. Information regarding the laboratory requirements for detection limits, precision, and accuracy of pore water samples are summarized in Table 5, found in Subsection 2.10 of the SAP, and discussed in Subsection 2.10 of the SAP and the QAPP (Appendix A).

5.3. Rock Core Analysis

Twelve previously extracted rock cores will be used to obtain material for analysis for primary porosity by the fixed laboratory. The location of the rock core samples was recorded by the entity that collected them. The samples will be selected in order to bias core selection toward those with evidence of primary porosity. Stone will presume that matrix diffusion in rocks with little or no primary porosity is de minimis. Core selection will also be biased toward locations where relatively high dissolved concentrations have been noted. Information regarding the laboratory requirements for detection limits, precision, and accuracy of rock samples are summarized in Table 5, found in Subsection 2.10 of the SAP, and discussed in Subsection 2.10 of the SAP and the QAPP (Appendix A).

5.4. Comprehensive Groundwater Sampling

Groundwater samples will be collected from up to 50 groundwater monitoring wells for one or more of the following reasons:

- Obtain representative total and dissolved metals samples from approximately 8 shallow overburden monitoring wells. These samples will be analyzed for total and dissolved TAL metals by USEPA Methods 6010/7470/7000;
- Obtain additional CVOC and metals results from select monitoring wells to support trend analysis. These samples will be analyzed for CVOCs via USEPA Method 8260B and select samples will be analyzed for TAL metals by USEPA Methods 6010/7470/7000; and/or

- Obtain current geochemical data to support the evaluation of natural attenuation in the dissolved groundwater plumes. These samples will be field screened for nitrate/nitrite, sulfate/sulfite, ferrous/ferric iron, manganous/manganic manganese, and chloride using a Hach field test kit, and analyzed by a fixed lab for VOCs via USEPA Method 8260B, ethane/ethene via Method RSK 175, and TOC via USEPA Method 9060A; a subset of 10 wells will also undergo microbial analysis for *Dehalcoccoides* spp. (qDHC) by proprietary methods. In addition, should the preceding qDHC analyses indicate that microbial degradation is occurring, additional TCE reductase (tceA), vinyl chloride reductase (bvcA), vinyl chloride reductase (vcrA) analyses will be performed by proprietary methods to identify specific biodegradation processes.

The monitoring wells selected for sampling are depicted on Figures 3a and 3b, and summarized in Table 3, found in Subsection 2.7.3.2 of the SAP. The sampling program for the 50 wells will be performed over an approximately 5-day period. Information regarding the laboratory requirements for groundwater metals, VOCs, and natural attenuation parameter samples regarding detection limits, precision and accuracy are summarized in Table 5, found in Subsection 2.10 of the SAP, and discussed in Subsection 2.10 of the SAP and the QAPP (Appendix A).

5.5. Residential Water Sampling

Representative groundwater samples will be collected from two private, non-potable water wells located north of the Property. Access to the wells has been granted for the first time. The wells are used solely for irrigation purposes, as the residences are supplied with drinking water by the Town of North Kingstown.

The data obtained from these tasks should be of sufficient quality to allow the project team to make decisions regarding potential impact to drinking water receptors. The screening levels used for this investigation are RIDEM groundwater standards and USEPA RSLs. These samples will be analyzed for low-concentration VOCs via USEPA Method 524.2.

The wells selected for sampling are depicted on Figures 3a and 3b, and summarized in Table 4, found in Subsection 2.8.3 of the SAP, and on SAP Figure 6. The sampling program for the private, non-potable water wells will be performed in a 1-day period. Information regarding the laboratory requirements for drinking water VOCs samples regarding detection limits, precision and accuracy are summarized in Table 5 found in Subsection 2.10 of the SAP, and discussed in Subsection 2.10 of the SAP and the QAPP (Appendix A)

5.6. Source Area Investigation

To further evaluate the western portion of the Source Area, a minimum of 13 soil core locations will be advanced along two, perpendicular transects with monitoring well EA-102D at the center (intersection) of the transects (Figure 7). One soil core will be advanced immediately adjacent to monitoring well EA-102D; the balance of the borings will be spaced 15-feet apart along each transect. The coring program will be expanded in a gridded pattern (with 15-ft spacing) as needed, based on the results from the initial two transects, to delineate the horizontal and vertical distribution of NAPL.

An additional 15 soil coring locations will be advanced within the remainder of the Source Area: five within the “core” of the Source Area, and five in the eastern portion of the Source Area in the vicinity of EA-119D, and an additional five to be determined based on the findings at the first 10 locations (Figure 7). The coring program will be expanded in a gridded pattern (with 30-ft spacing) to delineate the horizontal and vertical distribution of total target CVOCs with concentrations >10 ppm in soil.

Soil borings will be advanced to the top of bedrock, expected to be present between about 25 to 35 feet bgs, or until refusal is encountered. Soil cores will be collected in continuous 5-foot intervals, and soil samples will comprise a 0- to 1-ft depth interval, followed by 2-foot intervals measured from ground surface. Based on this sampling strategy, an estimated 15 soil samples will be collected from each soil boring. Each soil sample will be field-screened for soil headspace. Each soil sample that has a soil headspace photoionization detector (PID) reading greater than 100 ppm v/v will be field-screened for NAPL using "Oil Red O" dye. Each soil sample will be analyzed by the mobile laboratory for target VOCs via modified EPA Method 8260B. Aliquots of 10 selected soil samples will be further analyzed by a fixed laboratory for TPH (C_9 to C_{36} hydrocarbons) via EPA Method 8100 M. Sample selection for TPH analysis will be biased towards soils that exhibit the highest PID readings and or visual/olfactory evidence of contamination within the "smear zone". In addition, 10 selected soil samples (five from the sand/gravel unit and five from the basal till unit) will be collected for analysis of the following physical properties: bulk density and porosity by ASTM D7263, and fraction organic carbon (FOC) by ASTM D2974. Information regarding the laboratory requirements for detection limits, precision, and accuracy of soil samples are summarized in Table 4, found in Subsection 2.10 of the SAP, and further discussed in Subsection 2.10 of the SAP and the QAPP (Appendix A).

Up to three NAPL monitoring wells will be installed in the vicinity of well EA-102D, should mobile NAPL be detected during the Source Area soil investigation described above. The proposed locations are unknown at this time, and will be based on the results of the Source Area soil investigation; their locations and screened intervals are considered critical decisions, subject to the approval of USACE and RIDEM, as described in Subsection 10.1 of the Work Plan. The general location of these wells will be in the western portion of the Source Area, which is depicted in Figure 7.

Up to three NAPL samples will be collected from wells to be installed in the vicinity of well EA-102D, should mobile NAPL be detected during the Source Area soil investigation described in Subsection 5.2. The proposed locations are unknown at this time, and will be based on the results of the Source Area soil investigation, and their locations and screened intervals are considered critical decisions, subject to the approval of USACE and RIDEM, as described in Subsection 10.1 of the Work Plan. The general location of these wells will be in the western portion of the Source Area, which is depicted in Figure 7. Information regarding the laboratory requirements for NAPL samples regarding detection limits, precision and accuracy are summarized in Table 5, found in Subsection 2.10 of the SAP, and discussed in Subsection 2.10 and the QAPP (Appendix A).

5.7. Shallow Groundwater Assessment at the North Kingstown DPW Facility

Two shallow overburden groundwater monitoring wells will be installed at the North Kingstown DPW property, in the vicinity of the North Kingstown DPW Building, as depicted on Figure 9. Due to seasonal groundwater fluctuations of up to 9 ft, the screened interval of the groundwater monitoring wells to be installed at the North Kingstown DPW property will be 15 ft, set to straddle the top of the unconfined shallow groundwater table, with 3 ft above and 12 ft below the groundwater elevation observed in the borehole at the time of the investigation, which will be following the seasonal high. This procedure includes only field screening of soil headspace.

Groundwater samples will be collected from two shallow overburden groundwater monitoring wells to be installed at the North Kingstown DPW Property, in the vicinity of the North Kingstown DPW Building, as depicted on Figure 9. In addition, a soil sample will be collected from the depth interval with the highest soil headspace readings, or the water table (if no soil headspace readings are elevated), and analyzed for a focused list of VOCs by the mobile laboratory, in order to compare elevated soil VOC headspace readings to soil VOC

concentrations. The groundwater sampling program for these wells will be executed in a single day. Information regarding the laboratory requirements for groundwater VOCs samples regarding detection limits, precision and accuracy are summarized in Table 4 and discussed in Subsection 2.10 of the SAP and the QAPP (Appendix A).

5.8. Synchronous Groundwater Elevation Monitoring

Groundwater elevations will be measured from up to 50 groundwater monitoring wells, in addition to the 50 wells proposed for sampling in Subsection 5.4. The data will be collected synchronously, within 1 day, in accordance with the methodology described in Subsection 2.13 of the SAP.

5.9. Bedrock Description and Measurement

Stone will document the type of bedrock present and the orientation of observed fractures in a newly-exposed bedrock outcrop west of the Source Area (see Figure 2).The data will be collected in accordance with the methodology described in Subsection 2.17 of the SAP.

6. CHEMICAL ANALYSIS PROGRAM

6.1. Sample Analysis

Water, soil, and rock samples will be collected and sent to a laboratory for analysis as part of the RI/FS field program described in previous sections of the Work Plan. Water samples will be collected from monitoring wells (Subsections 5.4, 5.5, 5.6, and 5.7), and will include pore water and surface water (Subsection 5.1). In addition, representative samples of water pumped from the wells during sampling—investigation-derived waste (IDW)—will be collected and sent to the laboratory for analysis. Soil samples will include sediment collected from wetlands or surface water bodies (Subsection 5.1) and soil from soil cores (Subsections 5.5 and 5.6). In addition, samples will be collected from soil IDW (e.g., drill cuttings). Rock samples will comprise existing rock cores submitted for porosity analysis (Subsection 5.3).

Certain samples will be submitted to an onsite laboratory (groundwater and soil samples) for VOC analyses, and others will be submitted to a fixed, offsite laboratory. The samples will be analyzed for the parameters and by the analytical methods listed below and in Table 4 of the SAP, found in SAP Subsection 2.10. Quality Assurance/Quality Control (QA/QC) samples comprising trip blanks, equipment blanks, field duplicates, and matrix spikes/matrix spike duplicates (MS/MSD) associated with the samples will also be collected and submitted for analysis as indicated in Table 5 of the SAP and described in detail in QAPP Worksheets # 12, 15, 20, 28.

6.2. Laboratory Methods and Reporting

Analytical methods, whether implemented by Stone's onsite laboratory or through a subcontracted, offsite laboratory under this contract are included in the descriptions of each RI/FS task in Subsections 5.1 through 5.5, as well as below in Subsections 6.2.2 and 6.2.3. The analytical methods are fully summarized, including proposed numbers of samples and QA/QC samples, in Table 5 of the SAP. The SAP also includes a summary of the limit of quantitation (LOQ) otherwise known as the laboratory reporting limits for each analytical method in QAPP Worksheets #15-A through 15-Y. Standards used to develop Project Action Levels include USEPA Region 3 Risk-Based Concentrations for tap water and residential and industrial soils, as well as freshwater screening benchmarks for surface water and sediment; and USEPA ecological soil screening levels (although none have been developed for the PCOCs at the Property and its vicinity). RIDEM groundwater quality standards for GA groundwater, and soil leachability criteria for GA and GB groundwater provide secondary "to be considered" screening concentrations. Details regarding each analytical method (i.e., DQOs, QA/QC acceptance criteria, etc.) are described in detail in in QAPP Worksheets # 12, 20 and 28.

6.2.1. Onsite Laboratory Methods and Reporting

6.2.1.1. Methods

Stone's onsite laboratory will use EPA Method 8260C and solid phase microextraction (SPME), ASTM D6520 as the introduction step to the gas chromatograph / mass spectrometer to analyze soil samples for the following target VOCs:

chloroform	1,1,2-trichloroethane	benzene
1,1-dichloroethane	1,1,2,2-tetrachloroethane	toluene
1,1-dichloroethene	tetrachloroethene	ethylbenzene
cis-1,2-dichlroethene	trichloroethene	total xylenes
trans-1,2-dichlroethene	vinyl chloride	1,2,4-trimethylbenzene

1,3,5-trimethylbenzene naphthalene

The abbreviated analyte list is being used to focus the Source Area investigation on the known and suspected COCs for the Additional Source Area Characterization, and to provide quicker analytical results. Additional information regarding the onsite laboratory's method is included in Subsection 2.10 and Table 5 of the SAP and in QAPP Worksheets # 12-A and 28-A.

In addition, although not associated with Stone's onsite laboratory, the groundwater natural attenuation parameters nitrate, nitrite, sulfate, sulfite, ferrous/ferric iron, and chloride will be measured in the field by Stone field personnel using Hach field test kits. The field analyses will be subject to QA/QC standards, as described in QAPP Worksheets #12-B and 20.

6.2.1.2. Reporting

Non-detect results for all parameters will be reported at the laboratory's limit of quantitation (LOQ), otherwise known as the reporting limit or practical quantitation limit. Any detection between the limit of detection (LOD) or the established method detection limit and the LOQ will be qualified with a "J" estimated value. Data reporting and validation will comply with the requirements of the *PWS*.

6.2.2. Fixed Laboratory Methods and Reporting

6.2.2.1. Methods

Stone will utilize a number of fixed laboratories to obtain the various analytical methods required for the RI/FS investigation. The proposed analytical methods, sample media, and fixed laboratories selected for the analyses are summarized in Table 2.

Table 2: Summary of Fixed Laboratory Analytical Methods.

Analytical Method	Sample Media	Laboratory
VOCs by EPA Method 8260B	Pore water, Surface water, Sediment, Soil, and Groundwater, IDW	ESS Laboratory
Hardness by EPA Method 6010	Surface water	ESS Laboratory
Total Organic Carbon by the Lloyd Kahn Method	Sediment	ESS Laboratory
SVOCs by EPA Method 8270C	Soil	ESS Laboratory
TPH by EPA Method 8100M	Soil	ESS Laboratory
Petroleum Fingerprint by EPA Method 8015M	Soil	ESS Laboratory
Fraction Organic Carbon by ASTM D2974	Soil	ESS Laboratory
Porosity & Density by ASTM D7263	Soil	ESS Laboratory
Pesticides by EPA Method 8081A	IDW	ESS Laboratory
Polychlorinated biphenyls by EPA Method 8082	NAPL, IDW	ESS Laboratory
Viscosity by ATSM D445	NAPL	ESS Laboratory
Specific Gravity by ASTM D1298	NAPL	ESS Laboratory
Total Metals by EPA Methods 6010/7470/7000	Pore water, Surface water,	ESS Laboratory

Analytical Method	Sample Media	Laboratory
	Groundwater, IDW	
Dissolved Metals by EPA Methods 6010/7470/7000	Pore water, Surface water, Groundwater	ESS Laboratory
Microbial analysis	Groundwater	Microbial Insight
Porosity by API RP-40	Rock	Core Laboratory

Additional information regarding the fixed laboratories' method is included in Subsection 2.10 and Table 5 of the SAP and in QAPP Worksheets # 12-C through 12-Y.

6.2.2.2. Reporting

Non-detect results for all parameters will be reported at the laboratory's LOQ. Any detection between the LOD and the LOQ will be qualified with a "J" estimated value.

Analytical data deliverables will be provided by the fixed laboratory within the contracted turnaround time from date of sample receipt at the laboratory, and by the onsite laboratory within 24 hours from the time of sample receipt if sample volume into the laboratory is as allocated.

The hard copy analytical data package will be equivalent to a Level 3A Data Package. Data deliverables for this package include: case narrative, final results with surrogate standard recoveries, MS/MSD results, Laboratory Control Samples (LCS), and blank summary results.

For the fixed laboratory, one electronic data deliverable (EDD) file will be generated as a staged EDD (SEDD) for processing per USACE procedures. The onsite laboratory will provide an EDD in a suitable Excel format that can be easily manipulated by the project team and a SEDD format. All electronic data submitted by the contract laboratories are required to be error-free and in complete agreement with the hard copy data. The electronic CD of the data must be submitted with a transmittal letter from the laboratories that certifies that the file is in agreement with hard copy data reports and has been found to be free of errors.

The hard copy data package will include the information summarized below:

- Cover page including title of report, name and location of laboratory, contact information, name and location of any subcontractor laboratories, contract number unique ID for the report, client name and address, project name and location, statement of authenticity and signature/title of person releasing report, and total number of report pages;
- Table of contents;
- Case narrative (see description below);
- Cross reference of field sample IDs and laboratory IDs with associated test methods;
- Method numbers and summary;
- Chain-of-custody documentation including shipping documents;
- Sample receipt checklist;
- Telephone conversation and e-mail records for the project;
- Dates of sample extraction and analysis;

- Sample results for all neat and diluted runs, including units, matrix, qualifiers, dilution factors, and percent solids;
- Sample preparation information, including batch numbers;
- Raw data for initial and continuing calibrations;
- Method detection limits and method/practical quantitation limits;
- Results, true values, and/or acceptance limits for MS/MSDs, method or preparation/calibration blanks, LCSs, internal standard areas, surrogate spikes, interference checks, and serial dilutions.

The case narrative will include the client name, project name and number, date of issuance, and a discussion of any deviations from analytical strategy (e.g., samples received but not analyzed), sample receipt issues (e.g., incorrect preservatives, air bubbles in VOC vials), missed holding times, definition of data qualifiers or flags, identification of manually-integrated data, technical problems, and QC failures or non-conformances and associated corrective actions. The report will be signed by the Laboratory Project Manager.

7. QUALITY CONTROL, DATA MANAGEMENT, AND DATA EVALUATION

7.1. Data Quality Objectives

Achieving the data quality objectives (DQOs) for the RI/FS field tasks will enable proper data interpretation, confident updating of the CSM, and a sound technical basis from which to evaluate remedy options for the Property. A summary of the task-specific DQOs for each field task is provided in Table 3.

Table 3: Summary of Task-Specific Data Quality Objectives.

Task	Data Quality Objective(s)
Collect additional pore water, surface water, and sediment samples from Hall Creek and vicinity, and northern wetland, during annual low-flow period.	Schedule the sampling during annual low-flow period. Collect all samples according to the approved methodologies identified in the SAP. Document sample locations and depth intervals in accordance with the requirements of the SAP. The LOQs or LODs for all the chemical parameters must be less than the project's decision limits.
Analysis of existing rock core for primary porosity.	Achieve analytical reporting limit objectives.
Collection and analysis of groundwater samples from approximately 50 monitoring wells.	Identify appropriate monitoring wells for sampling. Perform groundwater sampling in accordance with low-flow methodology to obtain representative samples. The LOQs or LODs for all the chemical parameters must be less than the SLERA risk-based screening limits.
Additional Source Area characterization.	Delineate the extent of the potential NAPL source in three dimensions. Characterize the chemical and physical properties of the NAPL. Install NAPL monitoring wells at appropriate screened intervals to collect NAPL samples. The LOQs or LODs for all the chemical parameters must be less than the project's decision limits.
Shallow groundwater assessment at the North Kingstown DPW facility.	Install shallow overburden groundwater monitoring wells. Perform groundwater sampling in accordance with low-flow methodology to obtain representative samples. The LOQs or LODs for all the chemical parameters must be less than the project's decision limits
Groundwater assessment of private wells located north of the Property	Perform groundwater sampling in accordance with low-flow methodology to obtain representative samples. The LOQs or LODs for all the chemical parameters must be less than the project's decision limits

QAPP Worksheets # 11, 12-A through 12-Y, and 20 provide greater detail regarding task and analytical DQOs for each task. These details have been incorporated into the descriptions of the procedures for the field tasks described in Subsections 2.1 through 2.16 of the SAP.

7.2. Quality Control

Appropriate documentation of chemical quality control shall be provided whenever sampling or analysis for chemical constituents is required in order to meet the *PWS* objectives. Quality control for traditional soils, geophysical, or geotechnical testing shall also be included. The laboratories to be used by Stone will be accredited by the National Environmental Laboratory Accreditation Program (NELAP) and by DOD-ELAP ISO/IEC 17025:2005. Stone's onsite laboratory shall meet the requirements of EPA and RIDEM requirements. Stone's onsite laboratory is accredited under the Department of Defense's (DoD) Environmental Laboratory Accreditation Program (DoD-ELAP) ISO/IEC 17025;2005 for VOCs by Method 8260C (ASTM6520D preparation) for solids and waters , and complies with all requirements of the *DoD Quality Systems Manual*, Final Version 4.2, dated October 2010 or more recent version.

7.2.1. Field Quality Control Audits

Field records will be reviewed by the field operations leader (FOL) to ensure that:

- Logbooks and standardized forms have been filled out completely and that the information recorded accurately reflects the activities that were performed.
- Records are legible and in accordance with good recordkeeping practices, e.g., entries are signed and dated, data are not obliterated, and changes are initialed, dated, and explained.
- Sample collection, handling, preservation, and storage procedures were conducted in accordance with the SAP and any deviations were documented and approved by the appropriate personnel.

7.2.2. Laboratory Quality Control Audits

Prior to being released as final, laboratory data will proceed through a tiered review process. Data verification starts with the analyst, who performs a 100% review of the data to ensure the work was done correctly the first time.

Following the completion of the initial verification by the analyst performing the data reduction, a systematic check of the data will be performed by an experienced peer or supervisor.

Laboratory data reduction procedures will be performed according to the following protocol. Information related to analysis will be documented in controlled laboratory logbooks, instrument printouts, or other approved forms. All entries that are not generated by an automated data system will be made neatly and legibly in permanent, waterproof ink. Information will not be erased or obliterated. Corrections will be made by drawing a single line through the error and entering the correct information adjacent to the strikeout. All changes will be initialed, dated, and, if appropriate, accompanied by a brief explanation. Unused pages or portions of pages will be crossed out to prevent future data entry. Analytical laboratory records will be reviewed by the Laboratory Quality Assurance (QA) Coordinator periodically, to verify adherence to documentation requirements.

7.2.3. Field and Analytical Quality Control

7.2.3.1. Field Quality Control

All field procedures performed throughout the RI/FS investigation will be performed pursuant to all Standard Operating Procedures (SOPs) and/or USACE-approved methods included in the SAP. All monitoring equipment used onsite will be maintained, calibrated, and operated as specified in the SAP.

Field QC samples will be collected during sampling to assess the accuracy and precision of the analytical data. These samples include trip blanks, equipment blanks, field duplicates, and matrix spike/matrix spike duplicates (MS/MSDs). The field QC samples and the acceptance criteria for these samples are summarized in Table 4.

Table 4: Summary of Field Quality Assurance Samples.

QA Sample	Description	Acceptance Criteria
Trip Blank	Trip blanks will be included with each shipment of VOC vials delivered to the Property. Trip blanks associated with aqueous VOC samples will originate in the laboratories (onsite and offsite) and will be prepared by filling two 40-mL VOA vials with laboratory deionized water and sealing the vials with septum-lined caps (allowing no headspace). Trip blanks will accompany the sample bottles to the sampling locations and will remain (unopened) in the shipping container until the sample bottles are delivered to the respective laboratories.	Analytes in the trip blanks, if detected, should be less than one-half the laboratory reporting limit.
Equipment Blank	Equipment blanks will be prepared by routing analyte-free water (provided by the laboratory) through or over non-dedicated sampling equipment after equipment decontamination and before field sample collection. Equipment blanks will be collected for samples collected with non-dedicated equipment (at a frequency of one per ten or less samples) and will be analyzed for the same VOC compound list as the primary samples.	Analytes in the equipment blanks, if detected, should be less than one-half the laboratory reporting limit.
Field Duplicate	Field duplicates will be collected at a frequency of one field duplicate for every 10 or less samples submitted for VOC analyses. Sample containers for aqueous field duplicates will be filled consecutively.	Field duplicate relative percent differences (RPDs) are considered acceptable if they are within +/- 30% for aqueous samples and within +/- 50% for solid samples.
MS/MSD	MS/MSD samples will be collected at a frequency of one for every 20 or less samples submitted for VOC analyses. For those samples designated as MS/MSDs and replicates, sufficient additional volume will be provided.	Acceptance criteria for the percent recoveries of spiked compounds and RPDs for the duplicate analyses will be specified in the QAPP

7.2.3.2. Analytical Quality Control

The selected analytical laboratories will have QC programs in place to ensure the reliability and validity of the analysis performed at the laboratories. All analytical procedures will be documented in writing as SOPs and each SOP will include the minimum requirements for the procedure. In general, the QC requirements include the following:

- Blanks (method, reagent/preparation, instrument)
- MS/MSDs
- Surrogate spikes
- Laboratory Control Samples (LCSs)
- Internal standard areas and retention times (GC/MS analysis)

The criteria by which these QC samples will be evaluated are specified in worksheets # 12-A through 12-O of the QAPP.

In order to demonstrate the accuracy of the onsite laboratory, three performance evaluation (PE) samples will be analyzed at the beginning of the field program. The PE samples will be purchased from Environmental Resource Associates (ERA). Two PE standards will be purchased: one with a concentration between 1 and 100 µg/Kg (low-range), and the second between 500 and 5,000 µg/Kg (high-range). The PE samples will include the limited analytes listed for field laboratory reporting listed in Subsection 6.2.2.1. The PE samples provided by ERA have performance limits published by the vendor. ERA will issue an acceptance criteria summary which is based on the lot number and the results from various labs.

7.2.4. Data Review and Validation

The completeness goal for the project is that more than 90% of the data for each environmental medium of interest for each major contaminant of concern must be usable (not qualified as "rejected"), with the exception of serial dilutions where the result for one dilution is rejected and replaced by that of another. Acceptable limits for laboratory quality control tests will be described in the laboratory-specific SOPs that will be part of the QAPP.

7.2.4.1. Fixed Laboratory Automated Data Review

One EDD will be provided by the fixed laboratory as a staged electronic data deliverable (SEDD) file in accordance with USACE requirements as described below.

Each fixed laboratory will report data using the SEDD format. The minimum requirement for the laboratories is the delivery of a SEDD Stage 2a deliverable, a PDF document file of the laboratories' final data report, and a hard copy of the laboratories' final data report. The laboratories will provide any supplemental information (e.g., chromatograms, instrument calibrations) upon request.

Once the SEDD electronic data files have been received from the laboratory, Stone will electronically review the files to check project data quality requirements using USACE Automated Data Review (ADR) software. The ADR Software will be provided by Laboratory Data Consultants Inc. through USACE. Stone will develop a comprehensive ADR project library file for all of the methods to be analyzed on the project. The project library files will accurately reflect all of the analytical quality requirements as documented in the final project QAPP and addendums and will be provided prior to field sampling. The project library file will be provided to the laboratory for use in screening the EDD submittals.

Each laboratory is required to check the integrity of the SEDD using the SEDD checker tool and check the SEDD file against the ADR project library using the Contract Compliance Screening (CCS) tool contained in ADR (Laboratory Version). EDD non-conformances generated by ADR for which the laboratory is responsible will be corrected at the laboratory. A new SEDD file will be resubmitted and reviewed again. The laboratory shall address via a brief explanation in the non-conformance log any non-conformance that is not within their control. The final SEDD extensible markup language (XML) file and final laboratory non-conformance report will be provided to USACE's chemist for review.

Stone will perform a data review on the SEDD XML file by importing the SEDD files directly into ADR. The files will be reviewed utilizing the ADR (Consultant Version) software to check for compliance using the same ADR Project Library used by the laboratory. The ADR reviewed files will be imported into Stone's copy of the USACE Environmental Data Management System (EDMS). Stone will provide USACE with:

1. The reviewed data from ADR in standard ADR format (A1 through A5) as .txt files; and

2. The USACE EDMS generated database file(s) in *.mdb format incorporating current chemistry data and corresponding field data via the USACE ftp website, or some other mutually agreed upon means.

The database file will be submitted to USACE after any subsequent updates. All postings to the USACE ftp website will be accompanied by notification of said posting to a USACE project point of contact.

All electronic data submitted by the laboratory is required to be error free, and in complete agreement with the hard copy data. Data files are to be delivered both by e-mail and on high-density CD accompanying the hard copy data reports. The disk must be submitted with a transmittal letter from the laboratory that certifies that the file is in agreement with hard copy data reports and has been found to be free of errors using the latest version of the ADR evaluation software provided to the laboratory. The laboratory will correct any errors identified by Stone or USACE.

Each laboratory is required to check the integrity of the SEDD using the Contract Compliance Screening (CCS) tool contained in ADR (Laboratory Version) against the ADR Project Library. EDD non-conformances generated by ADR that the laboratory is responsible for will be corrected at the laboratory and a new SEDD file will be resubmitted and reviewed again. The laboratory will address via a brief explanation in the non-conformance log any non-conformance that is not within their control. The data evaluation for this project will conform to the requirements summarized in Chapter 2 of EM 200 1 6 (USACE, 1997), as defined in the ADR project library. The data evaluation will include the following:

1. Review of chain-of-custody documents to verify sample identities.
2. Review of sample log-in documents to verify any potential problems with custody seals, container integrity, sample preservation, labeling, etc.
3. Review of field rinsate blank data to ascertain any potential problems with container contamination, preservative contamination, sampling equipment contamination, or cross-contamination between samples during transport.
4. Review of trip blank data to identify any potential problems with sample container contamination, preservative contamination, laboratory reagent water contamination, or cross-contamination between samples during transport.
5. Review of method blank data to determine the presence of any sources of contamination in the analytical process due to laboratory contamination.
6. Review the matrix spike (MS) data to evaluate the potential for matrix effects and as a measure of analytical accuracy. MS recoveries will be compared against laboratory acceptance criteria and the ADR project library acceptance criteria to determine if they are within or outside of warning and control limits for percent recoveries.
7. Review of matrix spike/matrix spike duplicate (MS/MSD) data to evaluate sample homogeneity and as a measure of analytical precision. MS/MSD data will be compared to laboratory acceptance criteria and the ADR project library acceptance criteria for the maximum relative percent deviation (RPD).
8. Review of any laboratory control sample and laboratory control sample duplicate (LCS/LCSD) data (if available) as a measure of analytical accuracy and precision. LCS/LCSD recoveries will be compared against laboratory acceptance criteria and the ADR project library to determine if they are within or outside of warning and control limits for percent recoveries.

9. Review of LCS/LCSD data (if available) as a measure of analytical precision. LCS/LCSD data will be compared to laboratory acceptance criteria for the maximum RPD.
10. Standard reference material (SRM) or Laboratory Control Sample (LCS) data (if available) as a measure of analytical accuracy. SRM and LCS data will be compared to the certified acceptable ranges of analytical values.
11. Review of sample and sample duplicate data (if available) as a measure of sample homogeneity and as a measure of analytical precision. Sample and sample duplicate data will be compared against the laboratory acceptance criteria and the ADR project library for the maximum RPD.
12. Review of surrogate recovery data to access extraction efficiency, effectiveness of sample introduction, and possible loss during cleanup activities. Surrogate recoveries will be compared to laboratory acceptance criteria and the ADR project library to determine if they are within or outside of acceptable limits.
13. Review of sample dates, extraction/digestion dates, and analysis dates to determine if maximum method-specific holding times were met or exceeded.
14. The following items would typically not be reviewed under a SEDD stage 2a data validation effort: instrument tunes, standard curves, internal standard recoveries, system performance check compound results, continuing calibration results, inter-element correction check results, laboratory notebook pages, calculations, etc. However, supporting data (i.e. calibration curves, instrument tuning information) will be stored at the laboratory for a minimum of three years.

7.2.4.2. Data Review by Onsite Laboratory

Following the completion of the initial verification by the analyst performing the data reduction and reporting in the field, a systematic check of the data will be performed by an experienced peer or supervisor. This check will be performed to ensure that initial review has been completed correctly and thoroughly and will include a review of:

- Adherence to the requested analytical method SOP;
- Correct interpretation of chromatograms, mass spectra, etc.;
- Correctness of numerical input when computer programs are used (checked randomly);
- Correct identification and quantification of constituents with appropriate qualifiers;
- Numerical correctness of calculations and formulas (checked randomly);
- Acceptability of QC data;
- Documentation that instruments were operating according to method specifications (calibrations, performance checks, etc.);
- Documentation of dilution factors, standard concentrations, etc.; and
- Sample holding time assessment.

7.2.5. Reporting on Quality Control

7.2.5.1. Field Corrective Action

Corrective action in the field may be needed when sampling procedures and/or field analytical procedures require modification due to unexpected conditions. The field team may identify the need for corrective action. The FOL will approve the corrective action and notify the Project Manager (PM). The PM, in consultation with the quality assurance officer (QAO), will approve the corrective measure. The FOL will ensure that the corrective measure is implemented by the field team.

Additions to the proposed SAP (i.e., more samples, additional sampling locations other than those specified in the SAP, etc.) are not considered corrective actions. However, these changes will be discussed with USACE and RIDEM during the weekly reports and before being implemented.

Corrective actions will be implemented and documented in the field record book or on a corrective action form. Documentation will include:

- A description of the circumstances that initiated the corrective action;
- The action taken in response;
- The final resolution; and
- Any necessary approvals.

No field crew member will initiate corrective action without prior review and authorization from the FOL.

7.2.5.2. Laboratory Corrective Action

Corrective action in the laboratory may occur prior to, during, and after initial analyses. A number of conditions such as broken sample containers, multiple phases, low/high pH readings, and potentially high concentration samples may be identified during sample log in or analysis. Following consultation with laboratory analysts and supervisory personnel, it may be necessary for the laboratory QA coordinator to approve the implementation of corrective action. If the non-conformance causes failure to achieve project objectives, the QAO or PM will be notified.

Laboratory corrective actions are performed prior to release of the data from the laboratory. The corrective action will be documented in both the laboratory's corrective action files, and in the narrative data report sent from the laboratory to the QAO. If the corrective action does not rectify the situation, the laboratory will contact the QAO, who will determine the action to be taken and inform the appropriate personnel.

8. FEASIBILITY STUDY

The results of the RI/FS supplemental investigation and the updating of risk assessments will provide the necessary data to complete an RI and FS for the Property. The FS will develop and screen remedial alternatives for the Property. An initial evaluation will be conducted to screen various technologies for each type of media at the Property. Technologies that cannot be implemented technically at the Property will be eliminated from considerations. The alternatives that are not eliminated during the initial screening phase will be carried forward to the detailed evaluation. The detailed analysis will further refine each remedial alterative using the categories as described below. The detailed analysis will provide the decision makers (USACE) with sufficient information to compare the alternatives with respect to each other and then select an appropriate remedy. This evaluation will provide a basis for USACE to select the preferred remedial alternative and to develop a Proposed Plan, followed by the selected remedy in a Decision Document.

The FS will focus on strategies that will address remedial action objectives developed in the FS, and will likely include the general categories of source treatment, management of migration, and natural attenuation.

Due to the complicated nature of the contaminant distribution and migration pathways, the FS will also consider combining remedial technologies/approaches rather than simply looking at one at a time. For example, combinations such as the following may be considered: unconsolidated deposits excavation and natural attenuation for bedrock contamination; hydraulic containment for Property boundary management of migration and natural attenuation for the off-Property plume; thermal treatment of on-Property contaminants and natural attenuation for the off-Property plume; and permeable reactive barrier wall for treatment of the unconsolidated deposits plume as it leaves the Property and natural attenuation for the off-Property plume.

The FS will follow the general process outlined in EPA's *Guidance for Conducting Remedial Investigations and Feasibility Studies under CERCLA – Interim Final* (USEPA, 1988a), and will consider the requirements of the RIDEM Remediation Regulations. Section 7.04 of RIDEM *Remediation Regulations,* dated November 2011 requires that at least two alternatives must be carried forth in the FS other than the no action/natural attenuation alternative. RIDEM has requested that the no action alternative be evaluated. Stone's December 21, 2010 Proposal for the project calls for a maximum of six (6) alternatives to be evaluated, which will accommodate RIDEM remediation regulations.

The following steps will be followed to complete the FS:

Step 1: Develop the Remedial Action Objectives (RAOs) for the Property; identify ARARs; establish allowable exposure based on HHRAs and ERAs; identify contaminants of concern (COCs) and Preliminary Remediation Goals (PRGs); and identify areas/media in need of remediation.

Step 2: Develop General Response Actions; determine remedial options to be evaluated; identify and screen remedial technology types and process options. Preliminary screening criteria will include effectiveness, implementability, and cost.

Step 3: Identify and develop potentially feasible remedial alternatives by assembling technology types and/or process options that are potentially feasible; evaluate alternatives in detail; and compare remedial alternatives to each other. The detailed analysis phase will include additional ARAR development and refinement of RAOs and PRGs as appropriate. Remedial alternatives will be evaluated against seven of the nine CERCLA evaluation criteria. The first two criteria are the threshold factors. Any alternative that does not satisfy both of these criteria is dropped from further consideration in the detailed analysis, unless the requisite basis for a waiver of an ARAR exists pursuant to CERCLA Section 121(d)(4). These are:

1. Overall protection of human health and the environment; and

2. Compliance with ARARs and TBCs.

Five primary balancing criteria and their sub categories are used to make comparisons between the remedial alternatives. Alternatives that satisfy the two threshold criteria are evaluated further using the following criteria:

1. Long-Term Effectiveness and Permanence;
    a. Overall protection of human health and the environment
    b. Compliance with ARARs
    c. Long-term effectiveness and permanence
    d. Reduction of toxicity, mobility, or volume through treatment
    e. Short-term effectiveness
2. Reduction of Toxicity, Mobility, and Volume Trough Treatment:
    a. Treatment process used and materials treated
    b. Amount of hazardous materials destroyed or treated
    c. Degree of expected reductions in toxicity, mobility, or volume
    d. Degree to which treatment is irreversible
    e. Type and quantity of residuals remaining after treatment
3. Short-Term Effectiveness;
    a. Protection of community during remedial actions
    b. Protection of workers during remedial actions
    c. Environmental impacts
    d. Time until remedial action objectives are achieved
4. Implementability;
    a. Technical feasibility
    b. Reliability of the technology
    c. Ease of undertaking additional remedial actions, if necessary
    d. Availability of services or materials
    e. Administrative feasibility
    f. Time required
    g. Potential threats
5. Cost.
    a. Capital costs
    b. Operating and maintenance costs
    c. Present worth costs

The remaining two criteria, State of Rhode Island acceptance and community acceptance, are modifying factors. State and community acceptance will be evaluated after receipt of comments on USACE's Proposed Plan, which will identify the remedial alternative preferred by USACE.

The FS Report, which will present the results of each of the steps described above, and will also include COC tables, human health PRG calculations, detailed ARARs analysis, and cost estimation details for the final, detailed alternatives. The proposed outline of the FS Report, as suggested in Table 6-5 of *Guidance for Conducting Remedial Investigations and Feasibility Studies under CERCLA – Interim Final*, is as follows:

- Executive Summary
- Introduction
- Identification and Screening of Technologies
- Development and Screening of Alternatives
- Detailed Analysis of Alternatives
- References

9. SCHEDULE AND DELIVERABLES

9.1. Project Schedule

The proposed schedule for the RI/FS fieldwork is summarized in Table 5:

Table 5: Proposed Feasibility Study Fieldwork Schedule

Task	Proposed Date(s)
Hall Creek/northern wetland Assessment	August 2013
Rock Core Analysis	July 2013
Additional Source Area Characterization	June-July 2013
Shallow Groundwater Assessment at the North Kingstown DPW Facility	June-July 2013
Groundwater Monitoring	July 2013
Private Well Water Sampling	July 2013

9.2. Project Deliverables

The deliverables for this task order include the following:

Table 6: Proposed Document Delivery Schedule

Task	Proposed Date(s)
Data Gaps Analysis Report	March 2013
Work Plan, including SAP, QAPP, combined APP/SSHSP/AHA	June 2013
Remedial Investigation Report	November 2013
Feasibility Study Report	November 2013
Proposed Plan document	March 2014
Decision Document	November 2014

9.2.1. Remedial Investigation / Feasibility Study Reports

A CERCLA-compliant Remedial Investigation Report and Feasibility Study Report will be prepared following the RI/FS fieldwork. The reports shall be standalone documents submitted at the same time and contain:

- An executive summary, body, conclusions, recommendations, and reference sections.
- A detailed description of the field investigation efforts.
- Maps showing investigated areas and contaminant results.
- Maps showing field water quality trends, including: TCE, total CVOCs, total petroleum VOCs, standard hydrogen electrode-corrected oxidation-reduction potential (SHE-corrected ORP), specific conductance, and approximate groundwater elevations.
- A minimum of two cross-sections illustrating the primary soil and groundwater contaminant distribution, lithology, and approximate groundwater elevations.
- A minimum of two cross-sections illustrating trends in SHE-corrected ORP, specific conductance, and approximate groundwater elevations.

- Logs of soil, rock, and well installation data results containing chemical and geotechnical data, lithology, and surveyed location and elevation.
- A table in the body of the report summarizing detected contaminants and field water quality results at the time of sampling (including SHE-corrected ORP and specific conductance).
- A description of the disposition of solid and liquid investigation-derived wastes (IDW).
- Supporting appendices documenting data collection, equipment calibration, and chemical analyses, and a chemical analysis quality evaluation report.
- A data CD or DVD containing the report and all raw and processed field data files—including laboratory data—in PDF (version 6 or later) and Excel/ASCII format. Data files shall be categorized using clearly labeled sub-folders. Scanned copies of field notes taken in conjunction, or in lieu of, field forms shall also be included. A README file shall accompany the data explaining the database architecture, steps for successful importing, and positional data.

The reports shall, at a minimum, include evaluations of the following items:

- An update of the CSM, as may be appropriate to reflect new findings regarding Property geology, hydrogeology, and water chemistry.
- The nature and extent of TCE and other COCs across the Property and its vicinity.
- The revised risk assessments.
- Identification of which source areas or other areas require remediation.
- Evaluation of a minimum of three separate remedial technologies.
- A recommended remedial technology.

All maps shall be completed in accordance with USACE (USACE, 2002a, 2001a) and USGS (Hansen, 1996) standards. All cross-sections shall have internally and externally consistent interpretation across direct push and other sampling data sets; show vertical and horizontal scales and use the same scales for each section; define vertical exaggeration; use solid, dashed, dotted, and queried lines to reflect confidence in data interpretations; define all line types and patterns in a legend; label starting and ending compass quadrants; and note significant features, such as wells, potential source areas, ground surface topography, and ground features (e.g., utilities). Data sets overlain onto these cross-sections, such as speciated or total contaminant concentrations, etc., will be at the same vertical and horizontal scales as the sections.

All raw data, procedures, and equations used shall be presented and referenced where taken from the literature or the manufacturers manual.

Drilling and well construction and well development data shall be entered into a database program, and shall be transmitted in its native file format onto the DVD. Data files will be categorized using clearly labeled sub-folders. A README file shall accompany the data, explaining database organization, access, positional data and associated errors, field screening results, and formal observations. In the case of laboratory data, the original laboratory report shall accompany the data.

Utility and subsurface site clearance geophysics investigations will include raw and processed data files. These will be categorized using clearly labeled sub-folders, with final e-copy (PDF from original report, not scanned) documenting all phases of data collection, processing, and interpretation, and SOW used by the geophysics subcontractor. Scanned copies of field notes taken in conjunction with, or in lieu of, field forms will also be included. A README file will accompany the data, explaining positional and geophysical data integration. A

location map in native file and PDF formats will accompany the data set, showing all investigation activity locations generating data contained on the DVD (scale, elevation datum, north arrow, labeled locations, etc., will be shown).

Requirements for laboratory analytical reporting are provided in Section 6.0.

9.2.2. Project Repository and Administrative Record

Stone will ensure that the multimedia (i.e., both paper and electronic format) project repository and/or administrative record is kept up to date of all "project-related information" to ensure that pertinent documentation and data are available for project reviews, and to provide a clear record of the performance-based contract (PBC) approach to support final decisions and phase completion. This repository is the property of USACE and will be made available to USACE upon request.

"Project-related information" includes all documents, including critical e-mails, generated during the course of this contract. These documents are expected to be maintained in both electronic and paper copies.

Stone will also update the repositories for the administrative record/project repository for FUDS activities which is established at QDC's office, located at 95 Cripe Street in North Kingstown, RI. The project repository and administrative record will be updated by Stone, and made available to the public, for the duration of the contract.

9.2.3. Instructions for Submittals of Electronics Documents to the FUDS Records Database/ Deliverable Requirements

All project-related documents provided to USACE in electronic format are to be delivered in the Adobe PDF format 1.4 (compatible with Acrobat 5 .0) with searchable text using the original image or other electronic document which is pre-approved by USACE. Files printed from scanned documents shall be as legible as the originals. Care shall be taken to optimize the scanning settings to avoid excessive PDF file size. All scanned documents shall be assembled into the fewest number of individual files as possible and grouped according to project task. When contracting this work, the PDF file generation settings need to be submitted in writing and approved by the government.

10. PROJECT MANAGEMENT

10.1. Project Planning

Project planning includes the overall project management activities and the development of the SAP, QAPP, and SSHP. Project management will include keeping stakeholders informed of project status, existing or potential problems, and any changes that may be required to prudently manage the project and meet the needs of the project stakeholders and decision makers. Monthly status reports will be prepared and circulated to all stakeholders, which will summarize activities completed during the previous month, activities planned for the upcoming month, updates to the project schedule, and problems encountered and the resolution. Additionally, during the field program, weekly progress updates will be provided to all stakeholders via e-mail, which will include recently obtained field or laboratory data, field observations, and field decisions made along with the rationale for those decisions. For critical field decisions, conference calls will be held with all interested stakeholders (including USACE, RIDEM, QDC and EPA) before field decisions are made. The background and relevant data will be summarized and recommendations presented during the call. Documentation of the decision will be circulated to all parties following each critical decision conference call, including the rationale for the decision. Many of these critical field decisions are expected to be time critical, therefore timely conference calls and prompt decisions will be necessary.

10.2. Project Organization

10.2.1. US Army Corps of Engineers

The project is being performed for USACE, New England District. USACE will provide execution and technical oversight as well as administrative support and review. The contact information for USACE team members is provided in Table 7.

Table 7: USACE Team Members Contact Information

Title	Name	Phone Number	Email Address
Project Manager	Casey Haskell, P.G.	978-318-8398	Casey.haskell@usace.army.mil
Project Geologist	Tracy Dorgan, P.G.	978-318-8499	Tracy.h.dorgan@usace.army.mil
Project Chemist	David Lubianez	978-318-8311	David.J.Lubianez@usace.army.mil
Innovative Technologies	Dan Groher, PE	978-318-8404	Daniel.M.Groher@usace.army.mil
Health & Safety Officer	Sheila Harvey	978-318-8504	Sheila.Harvey@usace.army.mil
Human Health Risk Assessor	Cynthia Colquitt	978-318-8042	Cynthia.A.Colquitt@usace.army.mil
Environmental Resources and Ecological Risk Assessor	Mike Penko	978-318-8139	Michael.Penko@usace.army.mil

10.2.2. Rhode Island Department of Environmental Management

The project is being performed under the regulatory oversight of RIDEM. Contact information for the RIDEM Project Manager is:

Richard Gottlieb, P.E.
Rhode Island Department of Environmental Management
Office of Waste Management

235 Promenade Street
Providence, RI 02908-5767
401-222-2797x7138
Richard.gottlieb@dcm.ri.us

10.2.3. Other Stakeholders

The EPA coordinator for the adjacent NCBC Sites is:

Christine A.P. Williams
US EPA New England, Federal Facility Superfund Section
5 Post Office Square, Suite 100
Mailcode OSRR07-03
Boston, MA 02109-3912
(617) 918-1384
williams.christine@epa.gov

The US Navy contact for the adjacent NCBC is:

Jeff Dale
Remedial Project Manager
BRAC PMO Northeast
4911 South Broad Street
Building 679, Philadelphia Naval Business Center
Philadelphia, PA 19112

The Property is part of the Quonset Development Corporation development. The contact is:

Steven King
Quonset Development Corporation
Quonset Point/Davisville Industrial Park
95 Cripe Street
North Kingstown, RI 02852

(401) 295-0044 x243

10.2.4. Stone Environmental

Stone is the lead contractor that will be performing the RI/FS field activities. Key Stone personnel for tasks performed for the RI/FS field operations and their specific responsibilities are discussed below. Contact information for Stone team members is provided in Table 8.

Table 8: Stone Environmental, Inc. Team Members Contact Information

Title	Name	Responsibilities	Mobile Phone	Email Address
Program Manager	Seth Pitkin	Program management, technical review and support	802-249-8700	spitkin@stone-env.com
Project Manager	Joseph Schmidl, P.G., C.W.S.	Project management, technical hydrogeologic interpretation and review	802-477-3126	jschmidl@stone-env.com

Title	Name	Responsibilities	Mobile Phone	Email Address
Field Operations Leader	David Linari	Data collection	802-578-5817	dlinari@stone-env.com
Site Safety Officer	David Linari	Site safety	802-578-5817	dlinari@stone-env.com
QA Officer	Kim Watson	Data quality	802-249-7753	kwatson@stone-env.com
Health & Safety Officer	Kim Watson	Health and safety	802-249-7753	kwatson@stone-env.com

10.2.4.1. Program Manager

The Program Manager is responsible for overall management of contract and task order requirements with USACE. He will assure that the schedule is being met and that necessary company resources are available for performance of project tasks. He will also provide final editorial review for all technical documents submitted for this project.

10.2.4.2. Project Manager (PM)

The PM organizes the assigned project staff and initiates project planning and implementation activities at the Task Order level. The PM controls the budget and schedule, ensures that contract requirements are met, and is responsible for management of all field activities (including subcontractors), data management and interpretation, coordination with NAE, and other stakeholders, and preparation of project deliverables. The PM is responsible for ensuring that all project activities conform to USACE requirements and specifications, field data acquisition meets the DQOs, and each form/report is reviewed for accuracy and content.

10.2.4.3. Field Operations Leader (FOL)

The FOL will report directly to the PM and is responsible for desktop planning, equipment procurement, scheduling, subcontracting, and mobilization for the field program. The FOL also has overall responsibility for completion of all field activities in accordance with this SAP. Specific responsibilities of the FOL include:

- Coordinating activities in the field;
- Assigning specific duties to field team members;
- Mobilizing and demobilizing the field team, including the onsite laboratory and drilling equipment, as well as Stone subcontractors;
- Directing the activities of subcontractors in the field;
- Resolving any logistical problems that could potentially hinder field activities, such as equipment malfunctions or availability, or weather-dependent working conditions; and
- Coordinating with the QAO (see below) in implementing field quality control (QC) including issuance and tracking of measurement and test equipment; the proper labeling, handling, storage, shipping, and chain-of-custody procedures used at the time of sampling; and control and collection of all field documentation.

10.2.4.4. Quality Assurance Officer (QAO)

The QAO is responsible for the project quality assurance/quality control (QA/QC) in accordance with the SAP and appropriate management guidance. The QAO will ensure that the project field activity readiness review is

conducted; approve variances during field activities before work continues; approve, evaluate, and document the disposition of non-conformance reports (NCRs). The QAO will also oversee and approve any required project training, audit/surveillance plans, and subsequently supervise these activities. The QAO will be responsible for coordinating the analytical services, the acquisition and delivery of sample bottles to the Property, and validation of the analytical data.

10.2.4.5. Health & Safety Officer (HSO)

The HSO is responsible for ensuring that health and safety procedures designed to protect personnel are maintained throughout the field activities conducted for this project. This will be accomplished by strict adherence to the project-specific Site Safety and Health Plan (SSHP) and compliance with all NAE safety and health guidance and Stone's corporate health and safety plan. The HSO will also be responsible for the coordination and communication of health and safety issues for field personnel.

10.2.4.6. Site Safety Officer (SSO)

All field personnel are responsible for implementing the safety requirements specified in the SSHP for field activities. However, one person will be designated to serve as the SSO. The SSO will be onsite during all Stone activities. The SSO is responsible for enforcing the requirements of the SSHP once work begins. The SSO has the authority to correct, immediately, all situations where noncompliance with the SSHP is noted and to stop work in cases where an immediate danger is perceived. A full description of SSO responsibilities will be outlined in the SSHP.

10.2.4.7. Field Staff

The field staff, who will be made up of Stone staff and also possibly Watermark field staff (see subsection 9.1.3.10.1) report directly to the FOL. The Stone personnel responsible for operating drill rigs will hold applicable licenses. The responsibilities of the field team include:

- Collecting samples, conducting field measurements, and decontaminating sampling equipment according to documented procedures stated in the SAP;
- Ensuring that field instruments are properly operated, calibrated, and maintained, and that adequate documentation is kept for all instruments;
- Collecting the required QC samples and thoroughly documenting QC sample collection;
- Ensuring that field documentation and data are complete and accurate;
- Operating and maintaining drilling equipment in accordance with documented procedures stated in the SAP;
- Analyzing samples in the onsite laboratory in accordance with documented procedures stated in the SAP;
- Ensuring that all health and safety protocols are followed for Stone field activities, in accordance with the project specific SSHP; and
- Communicating any non-conformance or potential data quality issues to the FOL and QAO.

10.2.4.8. Subcontractors

Stone has or will retain subcontractors in order to complete the requirements of the project. A description of these subcontractors and their responsibilities is presented below. The subcontractors are as follows:

10.2.4.8.1. Field Staff

Additional field staff may be provided by Watermark out of their Lowell or Hanover, MA offices. Watermark is headquartered at 175 Cabot Street, Lowell, MA 01854.

10.2.4.8.2. Remediation Design Contractor

Remediation design services will be provided by:

Larry Pannell
Watermark
175 Cabot Street
Lowell, MA 01854

10.2.4.8.3. Risk Assessment Contractor

Risk assessment services will be provided by:

Anthony Rodolakis
AMEC
2 Robbins Road
Westford, MA 01886

Michael J. Murphy
AMEC Environment & Infrastructure, Inc.
107 Audubon Road
Wakefield, MA 01880

10.2.4.8.4. Laboratory

Fixed laboratory services are currently anticipated to be provided by:

Laurel Stoddard
ESS Laboratory, Inc.
185 Frances Avenue
Cranston, Rhode Island 02910

Jennifer Ericson
Microbial Insights
2340 Stock Creek Blvd.
Rockford, TN 37853-3044

Hugh Peek
Core Laboratory, LC
6316 Windfern Road
Houston, TX 77040

The final laboratory selected to perform the analyses will be selected based on anticipated sample analysis turn-around time, QA procedures, and cost. The final selected laboratory will be specified in the SAP.

10.2.4.8.5. Geophysics Contractor

A geophysical contractor will be engaged to perform utility clearance as necessary at each soil boring location, prior to drilling activities. Geophysical services will be provided by:

Jeffrey Reid, P.G.
Hager-Richter Geoscience, Inc.
8 Industrial Way - D10
Salem, New Hampshire 03079

10.2.4.8.6. Surveying Contractor

A Rhode Island-licensed surveyor will be used to survey monitoring well locations and elevations.

Wesley Grant
Environmental Planning and Surveying, Inc.
52 Dugway Bridge Road
West Kingstown, Rhode Island 02892

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Quonset Development Corporation (QDC), 2012a. *Newcomb Road Grading and Drainage Plan, Quonset Business Park*, Sheet C3.0.

QDC, 2012b. *Jones Road Roadway and Infrastructure Improvements, Quonset Business Park*, 8 Sheets.

FIGURES

Figure 1 Locus Map

Figure 2 Project Area and Sample Locations

Figure 3a Proposed Monitoring Well Sampling Locations – North

Figure 3b Proposed Monitoring Well Sampling Locations – South

Figure 4 Historical Uses and Potential Sources of Contamination

Figure 5 Pore Water, Surface Water and Sediment Sample Locations

Figure 6a Shallow Overburden Groundwater Elevations on April 15, 2010

Figure 6b Deep Overburden Piezometric Head Elevations on April 15, 2010

Figure 6c Bedrock Piezometric Head Elevations on April 15, 2010

Figure 7 Proposed Source Area Soil Boring Locations

Figure 8 Contaminant Plume Maps

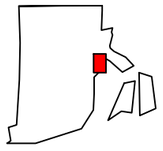
Figure 9 Proposed Well Locations, North Kingstown DPW Property

LEGEND

PR-58 Property Boundary

DATUMS

Coord. System: Rhode Island State Plane
Horizontal Datum: feet NAD 1983
Vertical Datum: feet NAVD 1988



Rhode Island

Note:

Source- orthophotography data collection flown April 29 through May 2, 2011. Rhode Island Geographic Information System(RIGIS, 2011).

REVISIONS

#	Date	Drwn	Chk'd	App'd	Description
1	10/26/12	DJL	JS	10/26/12	Displayed 2011 Orthos, Notes updated.

STONE ENVIRONMENTAL INC

TITLE: Locus Map

PROJECT: Former Nike Battery PR-58

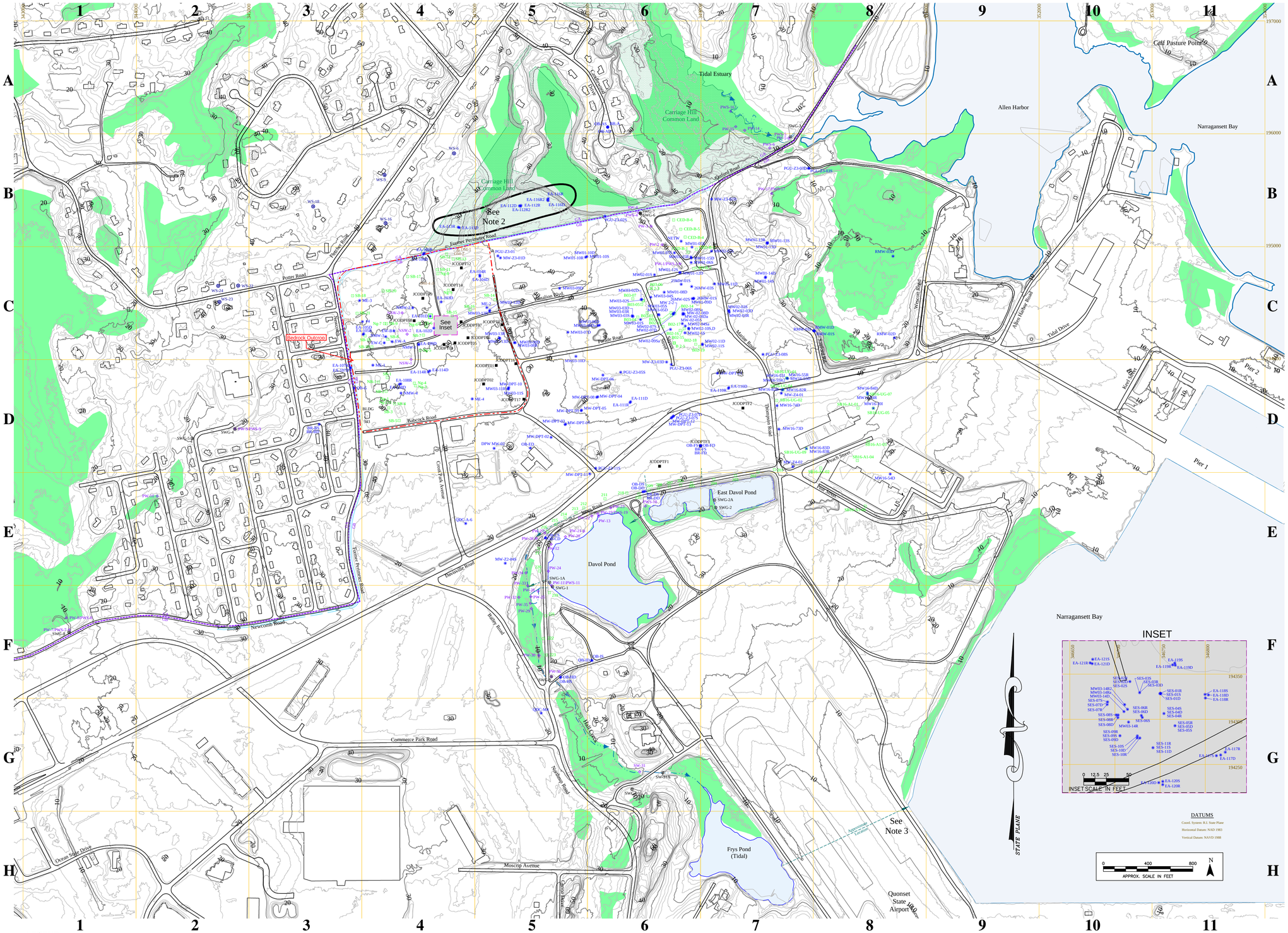
FIGURE NO.: 1

DATE: 10/26/12

DRAWN BY: DJL

CHECKED BY: JS

O:\PROJ-10\R\2305-R USACE NAE HTRW SB CONTRACT\01_USACE NIKE PR-58 FS IV, KINGSTOWN RIF-DATA\AUTOCAD



Monitoring Wells	Map Grid	Monitoring Wells	Map Grid	Monitoring Wells	Map Grid	Monitoring Wells	Map Grid	Monitoring Wells	Map Grid
29MW01S	C6	EA-117D	C4-I	MW01-15D	C6	MW03-10D	C5	MW-DPT-13	D6
29MW01S	C6	EA-117R	C4-I	MW01-15R	C6	MW03-11S	D5	MWZ-2-04S	E5
29MW02S	C6	EA-118S	C4-I	MW02-01S	C6	MW03-11R	D5	MW-Z3-01D	C5
29MW03S	C6	EA-118D	C4-I	MW02-02S	C7	MW03-12D	C5	MW-Z3-02D	B7
BR-A	A6	EA-118R	C4-I	MW02-03S	C7	MW03-12R	C5	MW-Z3-03D	D6
BR-B	D3	EA-119S	C4-I	MW02-03D	C7	MW03-13D	C5	MW-Z4-01	D7
BR-D	E6	EA-119D	C4-I	MW02-03R	C7	MW03-13R	C5	MW-Z4-02	D7
BR-F	D7	EA-119R	C4-I	MW02-04S	C6	MW03-14D	C4-I	NETW	B6
DPWMW02	C6	EA-120S	C4-I	MW02-04Sa	C6	MW03-14R	C4-I	NMW-1	C4
EA-101D	C4	EA-120D	C4-I	MW02-05S	C6	MW03-14Ra	C4-I	NMW-2	C4
EA-102D	C4	EA-120R	C4-I	MW02-06S	C6	MW03-14R2	C4-I	NMW-3	C4
EA-103D	C4	EA-121S	C4-I	MW02-07S	C6	MW16-10I	D8	NMW-4	D4
EA-104D	C5	EA-121D	C4-I	MW02-07D	C6	MW16-54D	E8	OB-A-S	A6
EA-104R	C5	EA-121R	C4-I	MW02-08S	C6	MW16-55D	D7	OB-A-D	A6
EA-105D	C4	BW-A	C4	MW02-08Sa	C6	MW16-55I	D7	OB-CS	E5
EA-105R	C4	BW-B	C4	MW-02-08D	C6	MW16-55R	D7	OB-CD	E5
EA-106D	C4	BW-C	C4	MW-02-08Da	C6	MW16-59RD	D7	OB-DS	E8
EA-106R	C4	IW-1	B4	MW02-09S	C6	MW16-73D	D7	OB-DD	E8
EA-107D	D3	ME-1	C4	MW02-09Sa	C6	MW16-74D	D7	OB-ED	D5
EA-107R	D3	ME-2	C5	MW02-10S	C6	MW16-82D	D7	OB-FS	D6
EA-108D	D4	ME-3	C3	MW02-10D	C6	MW16-82R	D7	OB-FD	D7
EA-108R	D4	ME-4	D4	MW02-11S	C7	MW16-83D	D7	OB-G	D3
EA-109D	C4	MW01-06S	C5	MW02-11D	C7	MW16-83R	D7	OB-HS	F6
EA-110D	D7	MW01-06S	C5	MW03-01S	C6	MW16-84D	D8	OB-HD	F6
EA-110R	D7	MW01-07S	C8	MW03-02S	C6	MW16-84R	D8	OB-IS	F6
EA-111D	D6	MW01-08D	C5	MW03-02D	C6	MW2-2	C6	OB-ID	F6
EA-111R	D6	MW01-09D	C5	MW03-03S	C6	MW-DPT-01	D7	PGU-Z3-01	C5
EA-112D	B5	MW01-10S	C5	MW03-03D	C6	MW-DPT-02	D5	PGU-Z3-02S	B6
EA-112R	B5	MW01-10D	C5	MW03-03R	C6	MW-DPT-03	D5	PGU-Z3-02D	B7
EA-112R2	B5	MW01-10R	C6	MW03-04S	C6	MW-DPT-04	D6	PGU-Z3-03D	B7
EA-113D	B4	MW01-11D	C7	MW03-05S	C6	MW-DPT-05	D5	PGU-Z3-05S	D6
EA-113R	B4	MW01-12S	C5	MW03-05D	C6	MW-DPT-06	D6	PGU-Z3-06S	D6
EA-114D	D4	MW01-12D	C6	MW03-06D	C6	MW-DPT-07	D5	PGU-Z3-07S	D6
EA-114R	D4	MW01-13S	B7	MW03-06Da	C6	MW-DPT-08	D6	PGU-Z3-07D	D6
EA-116D	B5	MW01-13D	B7	MW03-07D	C5	MW-DPT-09	D6	PGU-Z3-08S	C7
EA-116R	B5	MW01-13R	B7	MW03-08D	C5	MW-DPT-10	D5	PGU-Z3-11S	D6
EA-116R2	B5	MW01-14S	C7	MW03-08R	C5	MW-DPT-11	E6	QDC-A-6	E4
EA-117S	C4-I	MW01-14D	C7	MW03-09D	C5	MW-DPT-12	D6	QDC-M4	G6

Profiling Locations	Map Grid	Porewater / Surface Water	Map Grid	Porewater / Surface Water	Map Grid	Soil Borings	Map Grid	Soil Borings	Map Grid	Soil Borings	Map Grid
JCODPT01	D5	PW-4/PWS-4	B6	PW-32	F5	203	E7	B02-17	C6	SB-5	D4
JCODPT02	D5	PW-5/PWS-5	B7	PW-33	F6	204	E7	B02-18	C6	SB-6	C4
JCODPT03	C5	PW-6	B7	PW-34	E5	205	E6	B02-19	C5	SB-7	C4
JCODPT04	C4	PW-7/PWS-7	F1	PW-35	F5	206	E6	B03-03	C6	SB-8	C4
JCODPT05	C4	PW-8/PWS-8	F1	PW-36	F5	207	E6	B03-04	C6	SB-9	C4
JCODPT06	C4	PW-9/PWS-9	D2	SWG-1	F5	208	E6	B03-05	C6	SB-10	C4
JCODPT07	C4	PW-10	E2	SWG-1A	E5	209	E6	B03-06	C6	SB-11	C4
JCODPT08	C4	PW-11/PWS-11	F5	SWG-2	E7	210	E6	B03-07	C5	SB-12	C4
JCODPT09	C4	PW-12	E5	SWG-2A	E7	211	E6	GED-B-1	C6	SB-13	C4
JCODPT12	C4	PW-13	E8	SWG-3	E7	212	E6	GED-B-2	C6	SB-14	C5
JCODPT14	C4	PW-14	A7	SWG-4	D2	213	E6	GED-B-3	C6	SB-15	C4
JCODPT16	D5	PW-15	A7	SWG-5	D2	214	E5	GED-B-4	B6	SB-16	C4
JCODPT17	D5	PW-16	A7	SWG-6	B6	215	E5	GED-B-5	B6	SB-17	C4
JCODPTF1	D6	PW-17/PWS-17	B7	SWG-7	B6	216	E5	GED-B-6	B6	SB-18	C3
JCODPTF2	D7	PW-18	E6	SWG-8	F1	217	E5	NB-1	D4	SB-19	C4
JCODPTF3	D7	PW-19/PWS-19	E6	SWG-9	F5	218	E6	Np-1	D4	SB-20	C4
Debris Monitoring		PW-20	E5	SWG-10	G6	219	F6	Np-2	D4	SB-21	C3
DM-1	C4	PW-21	E5	**Water Supply Wells**		220	E5	Np-3	D4	SB16-A1-01	D8
DM-2	C4	PW-22	E6	WS-6	B4	221	F6	Np-4	D4	SB16-A1-02	D8
DM-3	C3	PW-23	E8	WS-9	B4	222	F6	Np-5	C4	SB16-A1-03	D8
DM-4	C4	PW-24	E5	WS-16	B4	223	F5	Np-6	C4	SB16-A1-04	D8
Surface Water		PW-25	F6	WS-18	B3	B 2-1	C6	Np-7	C4	SB16-A1-06	E8
NSW-1	C4	PW-26	E5	WS-22	C2	B 2-3	C6	Np-8	C4	SB16-UG-01	D7
NSW-2	C4	PW-27	E5	WS-23	C2	B02-12	C6	Np-9	C4	SB16-UG-02	D7
NSW-3	C4	PW-28	F5	WS-24	C2	B02-13	C6	SB-1	D4	SB16-UG-04	D7
PW-1/PWS-1	C6	PW-29	F5	**Soil Borings**		B02-14	C6	SB-2	D4	SB16-UG-05	D8
PW-2	B6	PW-30	F5	201	D7	B02-15	C6	SB-3	D4	SB16-UG-07	D8
PW-3	B6	SW-31, 31A	G6	202	D7	B02-16	C6	SB-4	D4	SB16-UG-09	D7

Pond Road Plan & Profiles by Charles A. Maguire and Associates (CMA, 1951)

Final Closure and Post-Closure Plan for Peabody Clean Industries Hazardous Waste Tank Farm Quonset Point, Rhode Island (ERA, 1984)

Final Engineering Report, Quonset Point Confirmation Study by Sirrine Environmental Consultants (SEC, 1988)

Engineering Evaluation of Contamination at the Nike Battery Site PR-58 and Disaster Village Training Area Quonset Point Naval Air Stations, North Kingstown Rhode Island - Final Report Volumes I and II (M&E, 1994)

Final Interim Report of Site 03/Nike Source Area Characterization and Offsite Investigation by EA Engineering, Science, and Technology (EA, 1998b)

Revised Draft Final Study Areas 01 and 04 and IR Program Sites 02 and 03, Phase III Comprehensive Remedial Investigation by EA Engineering, Science, and Technology (EA, 1998a)

Draft Characterization of CVOC Contamination at the Former PR-58 Nike Site and Adjacent Navy NCBC Davisville Site 03 by EA Engineering, Science, and Technology (EA, 2001)

Report of Findings, Steam Enhanced Remediation Pilot Test at the Former PR-58 Nike Site by EA Engineering, Science, and Technology (EA, 2008)

Orthophotography and LiDAR Terrain Data Collection flow April 7-8, 2007, Rhode Island Formerly Used Defense Sites, Final Report by Sky Research, Inc. (Sky, 2008)

Environmental Geographic Information System Release 1.1 by Tetra Tech NUS, Inc. (TetraTech-NUS, 2008)

Survey and GPS data collection performed by the New England District of the United States Army Corps of Engineers as part of the 2007 Synoptic Groundwater Sampling Program.
-GPS data collection performed by the USACE NAE - 11/05/08 and 09/24/09
-Survey by Environmental Planning and Surveying, Inc. - 12/08, 11/09, and 04/10

Data Provided Electronically by the Quonset Point Development Company, Nike Site.dwg and bikepath.dwg, April 2011 (QDC, 2011)

LEGEND

- Profiling Location
- Soil Boring Location
- Debris Monitoring Location
- Pore Water/Surface Water Sampling Location
- Surface Water Gauge Location
- Monitoring Well Location
- Water Supply Well Location
- Former Nike Battery PR-58 Project Area
- Carriage Hill Association Common Land (Approximate from RI GIS, 2011)
- Rhode Island Department of Environmental Management GA/GB Groundwater Classification Boundary
- Culvert with Flow Direction Arrow
- Creeks with Flow Direction Arrow
- Wetland
- Building
- 10' Ground Surface Contour
- 2' Ground Surface Contour
- Water Body
- Bike Path

WELL KEY

"S" wells are screened across water table or above LSC Silty-sand/silt/clay unit

"D" wells are screened below LSC Silty-sand/silt/clay unit and/or within five feet of the bedrock surface

"R" wells are open holes or screened in bedrock

NOTES

1) GA/GB Boundary as determined by Rhode Island Department of Environmental Management
-GA indicates area suitable for water supply usage
-GB indicates area not suitable for water supply usage
2) Wells located on Carriage Hill Association Common Land not accessible during the 2009-2010 Remedial Investigation.
3) Approximate location of concrete box culvert that outlets into Narragansett Bay from Frys Pond

REVISIONS	Date	Drwn	Chk'd	Appr'd	Description
1	10/29/12	DJL	JS	10/29/12	Added LiDAR Topography, building outlines, surface water displayed
2	12/14/12	DJL	JS	12/14/12	Added updated roads
3	1/31/13	DJL	JS	1/31/13	Darkened 10' contours, increased font size

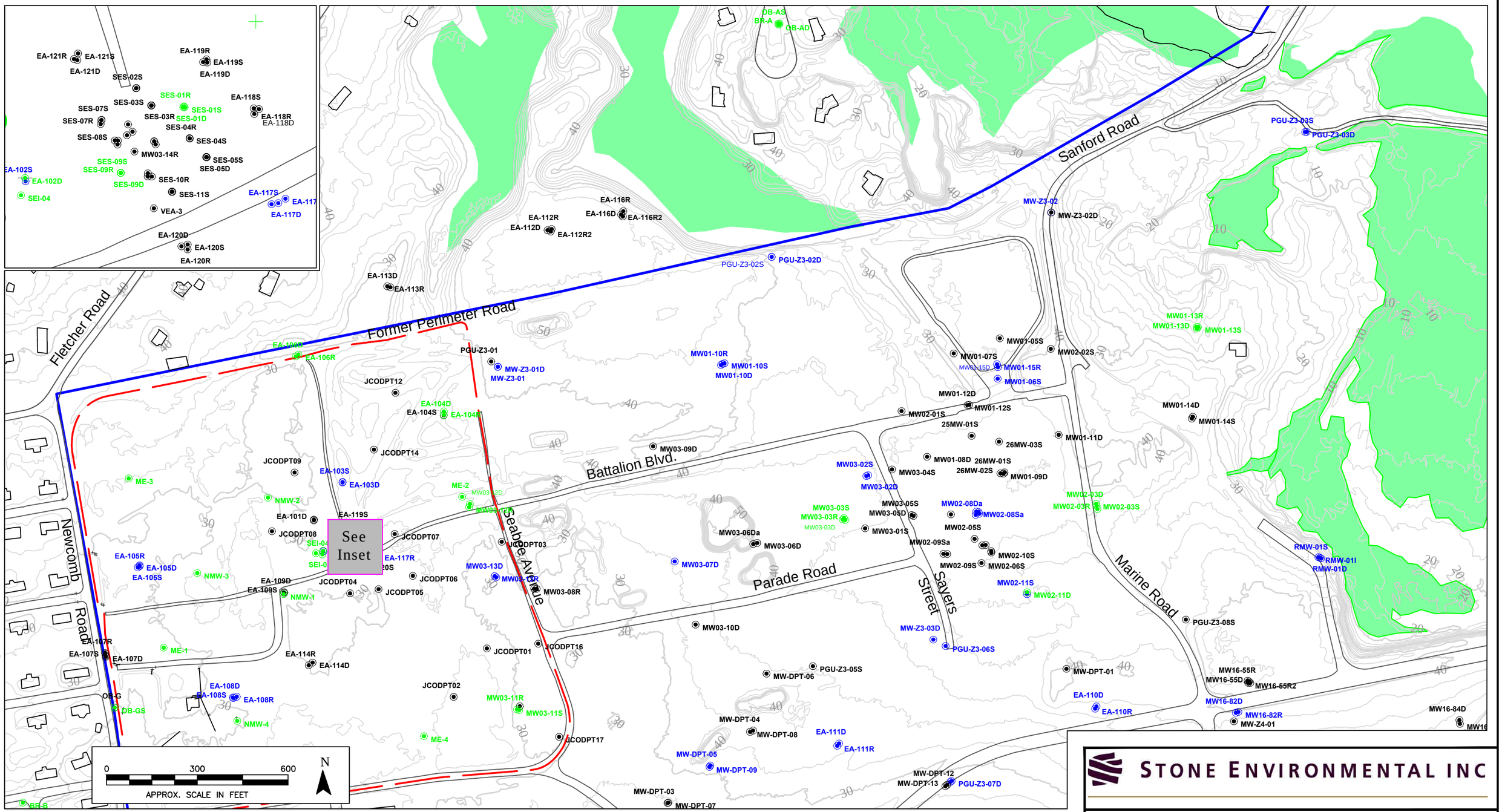
STONE ENVIRONMENTAL INC

TITLE: Property and Sample Locations

PROJECT: Former Nike Battery PR-58

FIGURE NO.: 2

DATE: 1/31/13 DRAWN BY: DJL CHECKED BY: JS



O:\PROJ\10JR\230S-R USACE\NAE\FRWS SB CONTRACT\03_USACE\NIKE PR-58\FS_N_KINGSTOWN RIF-DATA\AUTOCAD

LEGEND

- MW03-11R — Proposed Monitoring Wells to be Sampled
- MW03-11R — Proposed Monitoring Wells to Gauged
- EA-108D — Monitoring Wells
- PR-58 Property Boundary
- Wetlands
- Buildings
- Naval Construction Battalion Center Area
- GA/GB Groundwater Classification Boundary
- 10 — 10' Ground Surface Contour
- 2' Ground Surface Contour

DATUMS

Coord. System: Rhode Island State Plane
Horizontal Datum: feet NAD 1983
Vertical Datum: feet NAVD 1988

Notes:

Elevation Source: LiDAR terrain data collection flown April 7-8, 2007. Rhode Island formerly used defense sites (sky, 2008). Survey and GPS data collection performed by the New England District of the United States Army Corps of Engineers as part of the 2007 Synoptic Groundwater Sampling Program.
-GPS data collection performed by the USACE NAE - 11/05/08 and 09/24/09
-Survey by Environmental Planning and Surveying, Inc. - 12/08, 11/09, and 04/10
Data Provided Electronically by the Quonset Point Development Company, Nike Site.dwg and bikepath.dwg, April 2011 (QDC, 2011)

REVISIONS

#	Date	Drwn	Chk'd	App'd	Description
1	10/26/12	DJL	JS	10/26/12	Fixed Boundary, Show Buildings, Notes updated.
2	1/31/13	DJL	JS	1/31/13	darkened 10' contours, increased font size
3	3/13/13	DJL	JS	3/13/13	Changed Wells
4	9/13/13	SAH	JS	9/19/13	Identified gauged well, changed tables, legend, layout

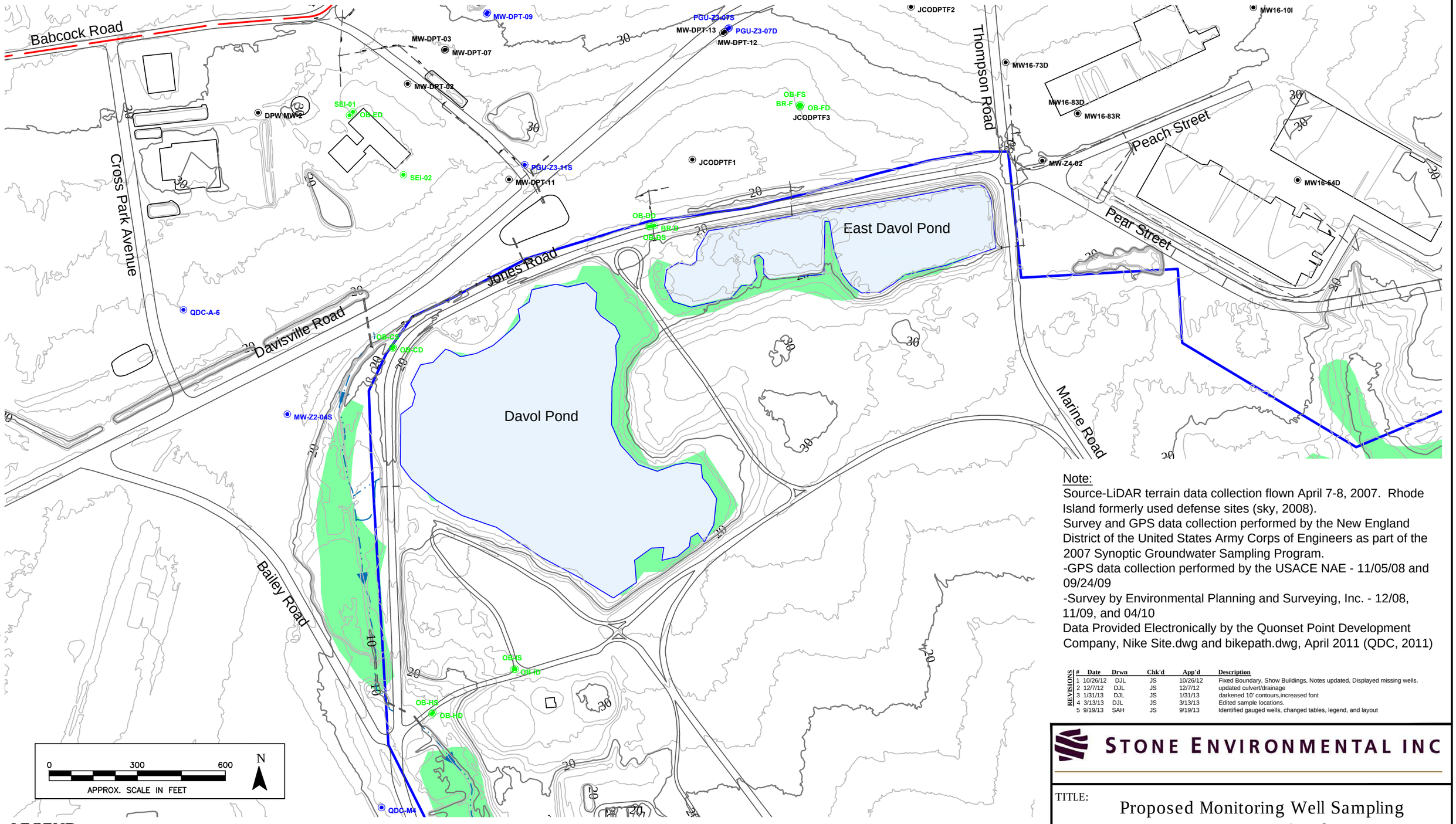
STONE ENVIRONMENTAL INC

TITLE: Proposed Monitoring Well Sampling Locations - North

PROJECT: Former Nike Battery PR-58

FIGURE NO.: 3a

DATE: 9/19/13 DRAWN BY: SAH CHECKED BY: JS



Note:
Source-LiDAR terrain data collection flown April 7-8, 2007. Rhode Island formerly used defense sites (sky, 2008).
Survey and GPS data collection performed by the New England District of the United States Army Corps of Engineers as part of the 2007 Synoptic Groundwater Sampling Program.
-GPS data collection performed by the USACE NAE - 11/05/08 and 09/24/09
-Survey by Environmental Planning and Surveying, Inc. - 12/08, 11/09, and 04/10
Data Provided Electronically by the Quonset Point Development Company, Nike Site.dwg and bikepath.dwg, April 2011 (QDC, 2011)

REVISIONS

#	Date	Drwn	Chk'd	App'd	Description
1	10/26/12	DJL	JS	10/26/12	Fixed Boundary, Show Buildings, Notes updated, Displayed missing wells.
2	12/7/12	DJL	JS	12/7/12	updated culvert/drainage
3	1/31/13	DJL	JS	1/31/13	darkened 10' contours,increased font
4	3/13/13	DJL	JS	3/13/13	Edited sample locations.
5	9/19/13	SAH	JS	9/19/13	Identified gauged wells, changed tables, legend, and layout

LEGEND

- MW03-11R — Proposed Monitoring Wells to be Sampled
- MW03-11R — Proposed Monitoring Wells to be Gauged
- EA-108D — Monitoring Wells
- PR-58 Property Boundary
- Wetland
- Buildings
- Naval Construction Battalion Center Area
- Water body
- Culvert/Drainage
- 10 — 10' Ground Surface Contour
- 2' Ground Surface Contour

DATUMS

Coord. System: Rhode Island State Plane
Horizontal Datum: feet NAD 1983
Vertical Datum: feet NAVD 1988

STONE ENVIRONMENTAL INC

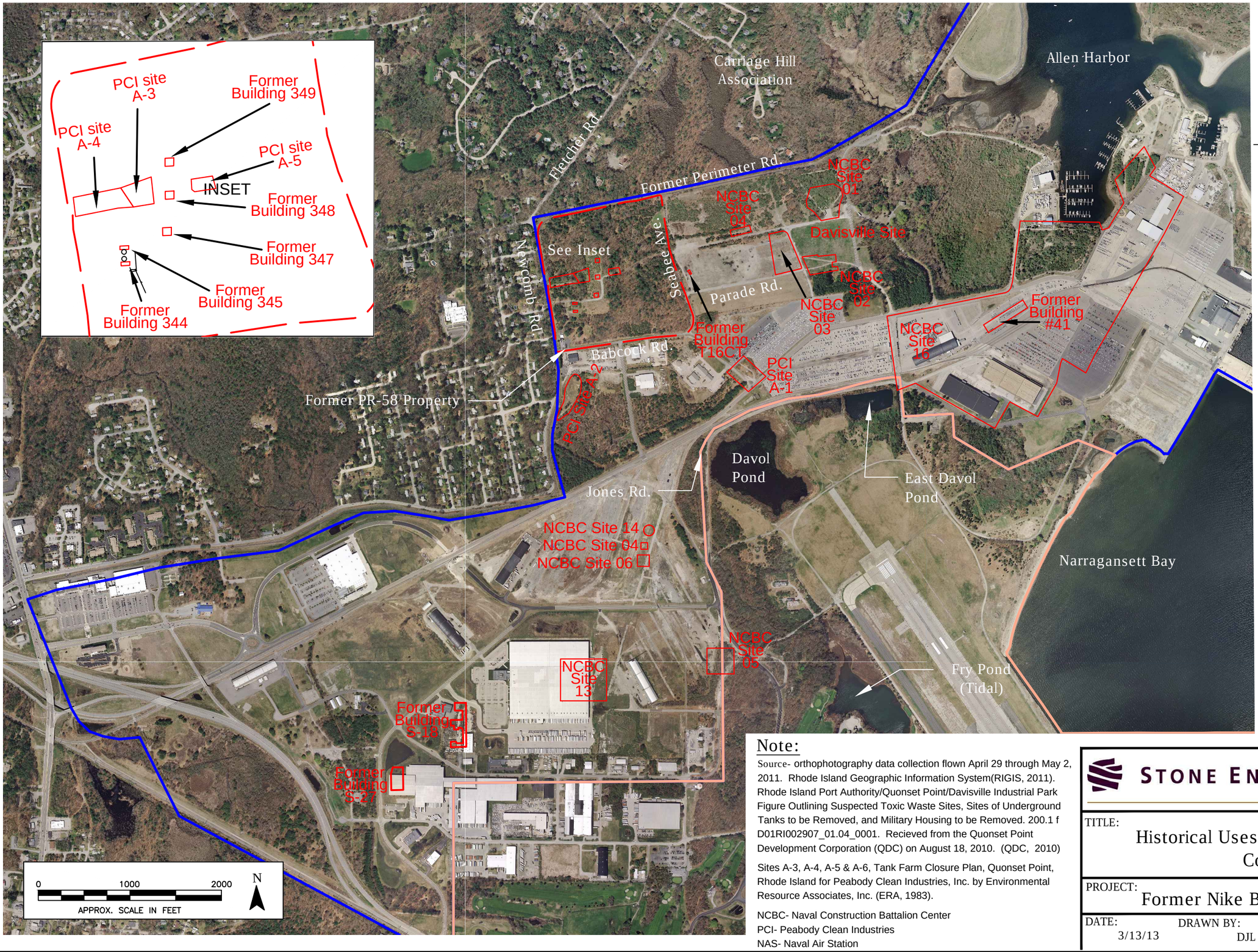
TITLE: Proposed Monitoring Well Sampling Locations - South

PROJECT: Former Nike Battery PR-58

FIGURE NO.: 3b

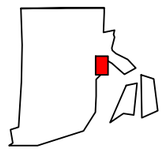
DATE: 9/19/13 DRAWN BY: SAH CHECKED BY: JS

O:\PROJ\10IR\2205-RI\SAC\ENAE\HTRW SIR CONTRACT\02\USACE NIKE PR-58\FS\N KINGSTOWN RIF\DATA\AUTOCAD



LEGEND

- PR-58 Property Boundary
- Site Boundary or Area of Interest
- Naval Construction Battalion Center Area
- Naval Air Station Area
- Former UST Location



Rhode Island

DATUMS

Coord. System: Rhode Island State Plane
Horizontal Datum: feet NAD 1983
Vertical Datum: feet NAVD 1988

REVISIONS

#	Date	Drwn	Chk'd	App'd	Description
1	10/26/12	DJL	JS	10/26/12	Displayed 2011 Orthos, Notes updated, inset added
2	3/13/13	DJL	JS	3/13/12	Edited PCI site names

Note:

Source- orthophotography data collection flown April 29 through May 2, 2011. Rhode Island Geographic Information System(RIGIS, 2011). Rhode Island Port Authority/Quonset Point/Davisville Industrial Park Figure Outlining Suspected Toxic Waste Sites, Sites of Underground Tanks to be Removed, and Military Housing to be Removed. 200.1 f D01RI002907_01.04_0001. Recieved from the Quonset Point Development Corporation (QDC) on August 18, 2010. (QDC, 2010)

Sites A-3, A-4, A-5 & A-6, Tank Farm Closure Plan, Quonset Point, Rhode Island for Peabody Clean Industries, Inc. by Environmental Resource Associates, Inc. (ERA, 1983).

NCBC- Naval Construction Battalion Center
PCI- Peabody Clean Industries
NAS- Naval Air Station

STONE ENVIRONMENTAL INC

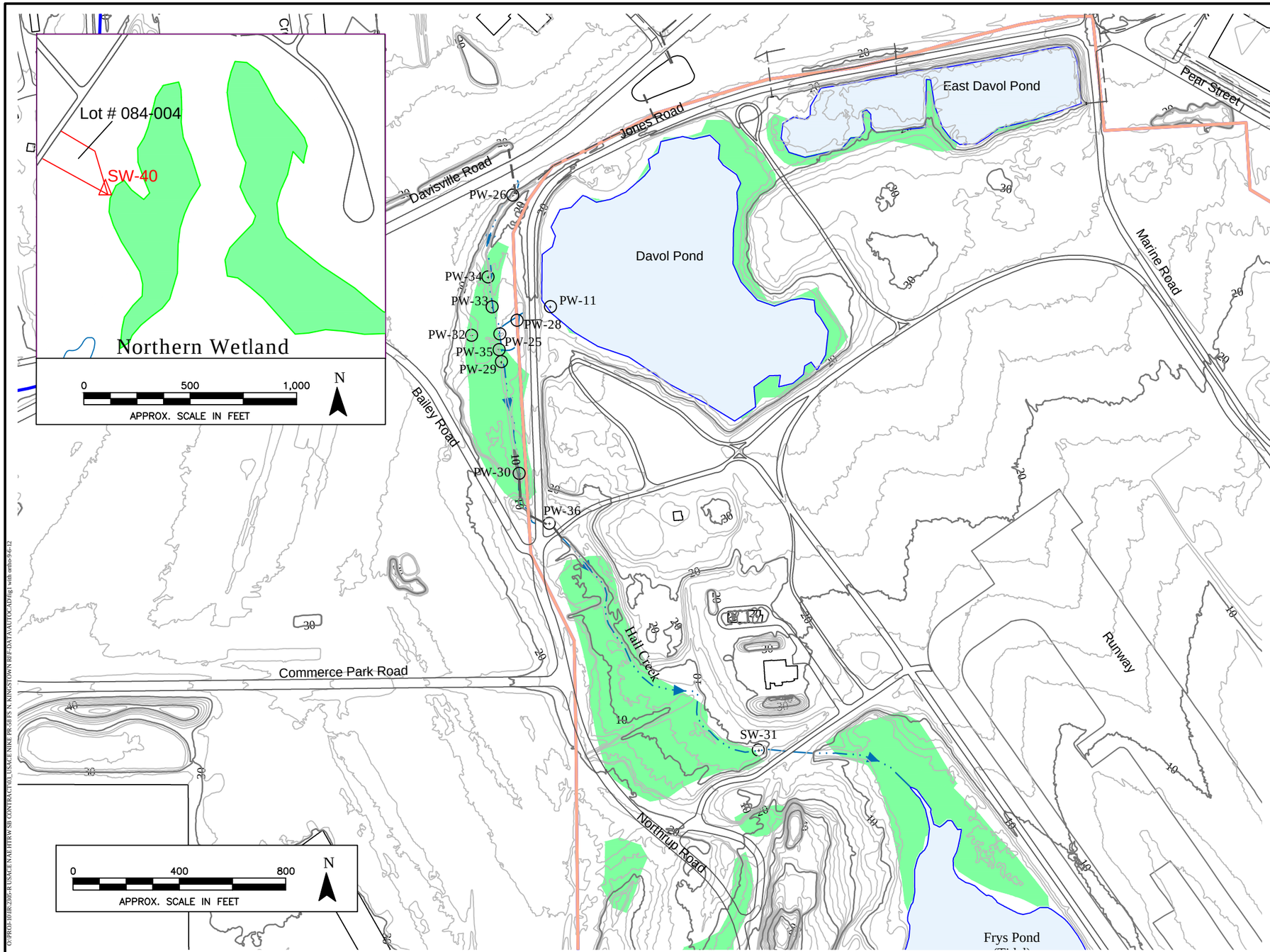
TITLE: Historical Uses and Potential Sources of Contamination

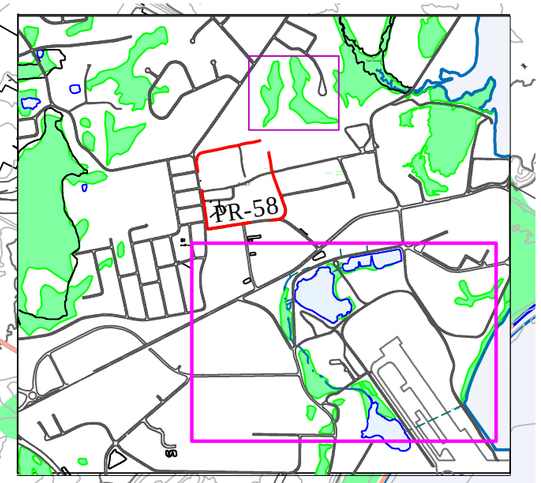
PROJECT: Former Nike Battery PR-58

FIGURE NO.: 4

DATE: 3/13/13 DRAWN BY: DJL CHECKED BY: JS

O:\PROJ-10\R\2305-R USACE NAE HTRW SB CONTRACT\03_USACE NIKE PR-58 FS N. KINGSTOWN RIF-DATA\AUTOCAD





Location Map

Note:

Source- LiDAR terrain data collection flown April 7-8, 2007. Rhode Island formerly used defense sites (sky, 2008).

Survey and GPS data collection performed by the New England District of the United States Army Corps of Engineers as part of the 2007 Synoptic Groundwater Sampling Program.

GPS data collection performed by the USACE NAE - 11/05/08 and 09/24/09

REVISIONS #	Date	Drwn	Chk'd	App'd	Description
1	10/27/12	DJL	JS	10/26/12	Displayed Topo, Show Buildings
2	12/7/12	DJL	JS	12/7/12	Notes updated, Displayed missing wells, updated culvert/drainage.
3	1/31/13	DJL	JS	1/31/13	darkened 10' contours,increased font, added northern sampling locations
4	3/18/13	DJL	JS	3/18/13	removed SW-41, and Lot ID

LEGEND

- PW-28 — Multimedia Sample Location
- SW-40 — Surface Water Sample
- Buildings
- Culvert/Drainage
- Stream with Flow Direction
- Wetland
- PR-58 Property Boundary
- Naval Construction Battalion Center Area
- Naval Air Station Area
- Water body
- Map Extent
- Map Inset
- 10 — 10' Ground Surface Contour
- 2' Ground Surface Contour
- Property lines

DATUMS

Coord. System: Rhode Island State Plane
Horizontal Datum: feet NAD 1983
Vertical Datum: feet NAVD 1988

STONE ENVIRONMENTAL INC

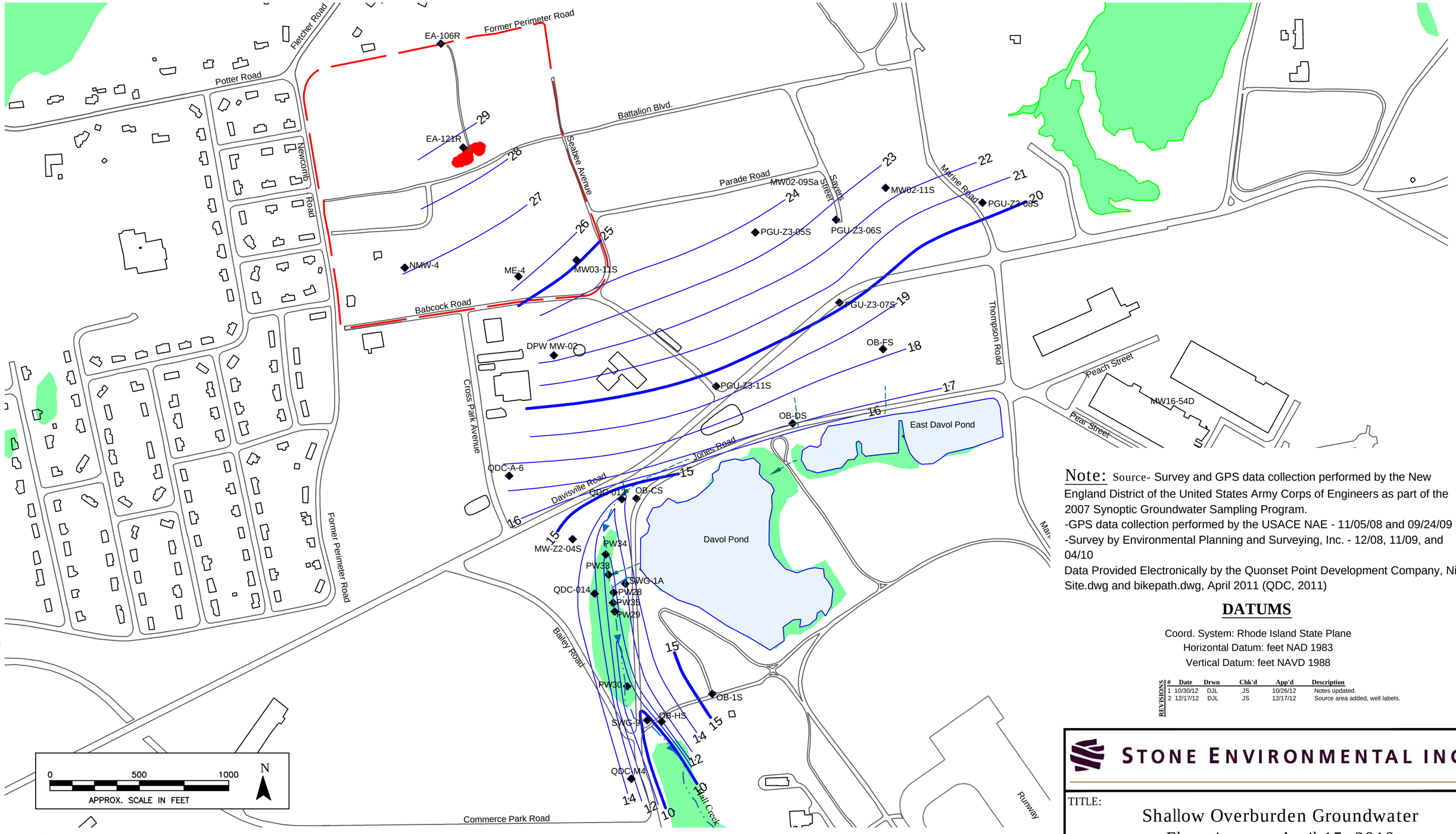
TITLE: Pore Water, Surface Water, and Sediment Sample Locations

PROJECT: Former Nike Battery PR-58

FIGURE NO.: 5

DATE: 3/18/13 DRAWN BY: DJL CHECKED BY: JS

O:\PROJ\0JR\2305-R USACE\NAE\FTRW\58 CONTRACT\03_USACE NIKE PR-58 FS N. KINGSTOWN RIF-DATA\AUTOCAD\fig1 with mlm-9-6-12



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Note: Source- Survey and GPS data collection performed by the New England District of the United States Army Corps of Engineers as part of the 2007 Synoptic Groundwater Sampling Program.
-GPS data collection performed by the USACE NAE - 11/05/08 and 09/24/09
-Survey by Environmental Planning and Surveying, Inc. - 12/08, 11/09, and 04/10
Data Provided Electronically by the Quonset Point Development Company, Nike Site.dwg and bikepath.dwg, April 2011 (QDC, 2011)

DATUMS

Coord. System: Rhode Island State Plane
Horizontal Datum: feet NAD 1983
Vertical Datum: feet NAVD 1988

REVISIONS

#	Date	Drwn	Chk'd	App'd	Description
1	10/30/12	DJL	JS	10/26/12	Notes updated.
2	12/17/12	DJL	JS	12/17/12	Source area added, well labels.

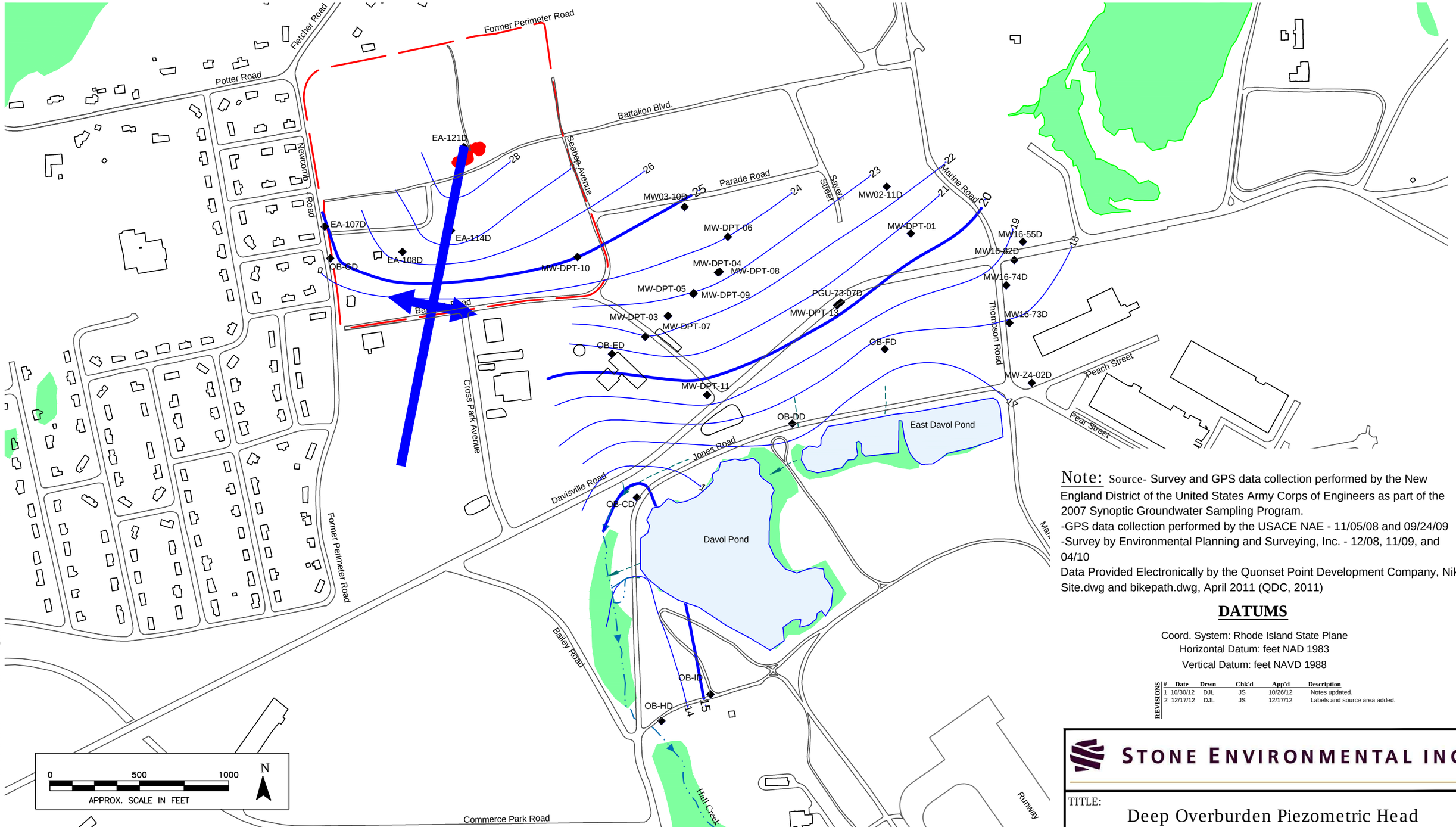
STONE ENVIRONMENTAL INC

TITLE:
Shallow Overburden Groundwater Elevations on April 15, 2010

PROJECT: Former Nike Battery PR-58

FIGURE NO.: 6a

DATE: 12/17/12 DRAWN BY: DJL CHECKED BY: JS



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LEGEND

- 1' Groundwater Elevation Contour
- 5' Groundwater Elevation Contour
- PR-58 Property Boundary
- Water Level Location
- Culvert
- Stream with Flow Direction
- Wetland
- Water body
- Buildings
- Source Area
- Groundwater Divide

Note: Source- Survey and GPS data collection performed by the New England District of the United States Army Corps of Engineers as part of the 2007 Synoptic Groundwater Sampling Program.
-GPS data collection performed by the USACE NAE - 11/05/08 and 09/24/09
-Survey by Environmental Planning and Surveying, Inc. - 12/08, 11/09, and 04/10
Data Provided Electronically by the Quonset Point Development Company, Nike Site.dwg and bikepath.dwg, April 2011 (QDC, 2011)

DATUMS

Coord. System: Rhode Island State Plane
Horizontal Datum: feet NAD 1983
Vertical Datum: feet NAVD 1988

REVISIONS

#	Date	Drwn	Chk'd	App'd	Description
1	10/30/12	DJL	JS	10/26/12	Notes updated.
2	12/17/12	DJL	JS	12/17/12	Labels and source area added.

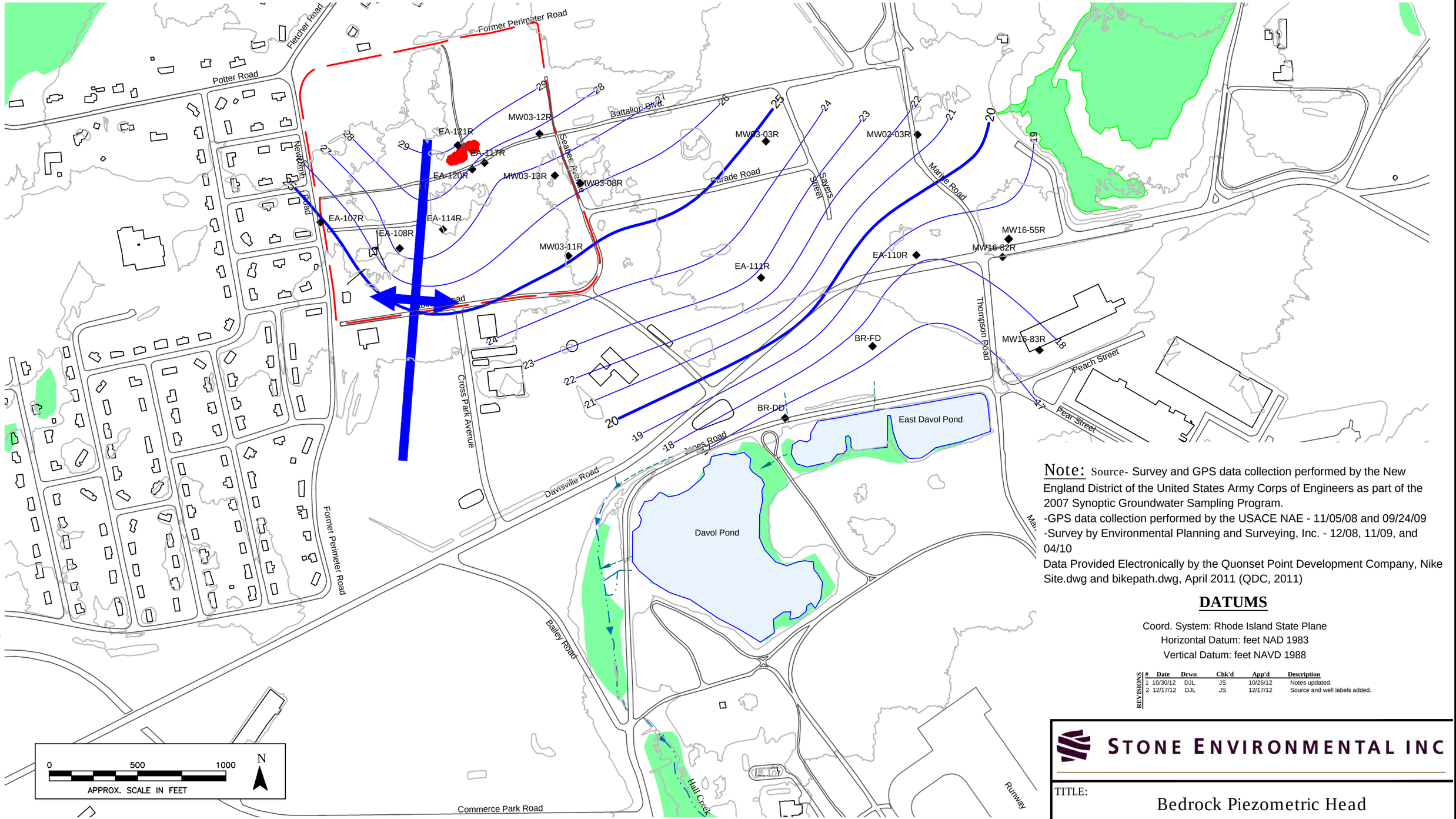
STONE ENVIRONMENTAL INC

TITLE: Deep Overburden Piezometric Head Elevations on April 15, 2010

PROJECT: Former Nike Battery PR-58

FIGURE NO.: 6b

DATE: 12/17/12 DRAWN BY: DJL CHECKED BY: JS



LEGEND

- 1' Groundwater Elevation Contour
- 5' Groundwater Elevation Contour
- PR-58 Property Boundary
- Water Level Location
- Culvert
- Stream with Flow Direction
- Wetland
- Water body
- Buildings
- Source Area
- Groundwater Divide

Note: Source- Survey and GPS data collection performed by the New England District of the United States Army Corps of Engineers as part of the 2007 Synoptic Groundwater Sampling Program.
-GPS data collection performed by the USACE NAE - 11/05/08 and 09/24/09
-Survey by Environmental Planning and Surveying, Inc. - 12/08, 11/09, and 04/10
Data Provided Electronically by the Quonset Point Development Company, Nike Site.dwg and bikepath.dwg, April 2011 (QDC, 2011)

DATUMS

Coord. System: Rhode Island State Plane
Horizontal Datum: feet NAD 1983
Vertical Datum: feet NAVD 1988

REVISIONS

#	Date	Drwn	Chk'd	App'd	Description
1	10/30/12	DJL	JS	10/26/12	Notes updated.
2	12/17/12	DJL	JS	12/17/12	Source and well labels added.

STONE ENVIRONMENTAL INC

TITLE:
Bedrock Piezometric Head Elevations on April 15, 2010

PROJECT: Former Nike Battery PR-58

FIGURE NO.: 6c

DATE: 12/17/12 DRAWN BY: DJL CHECKED BY: JS

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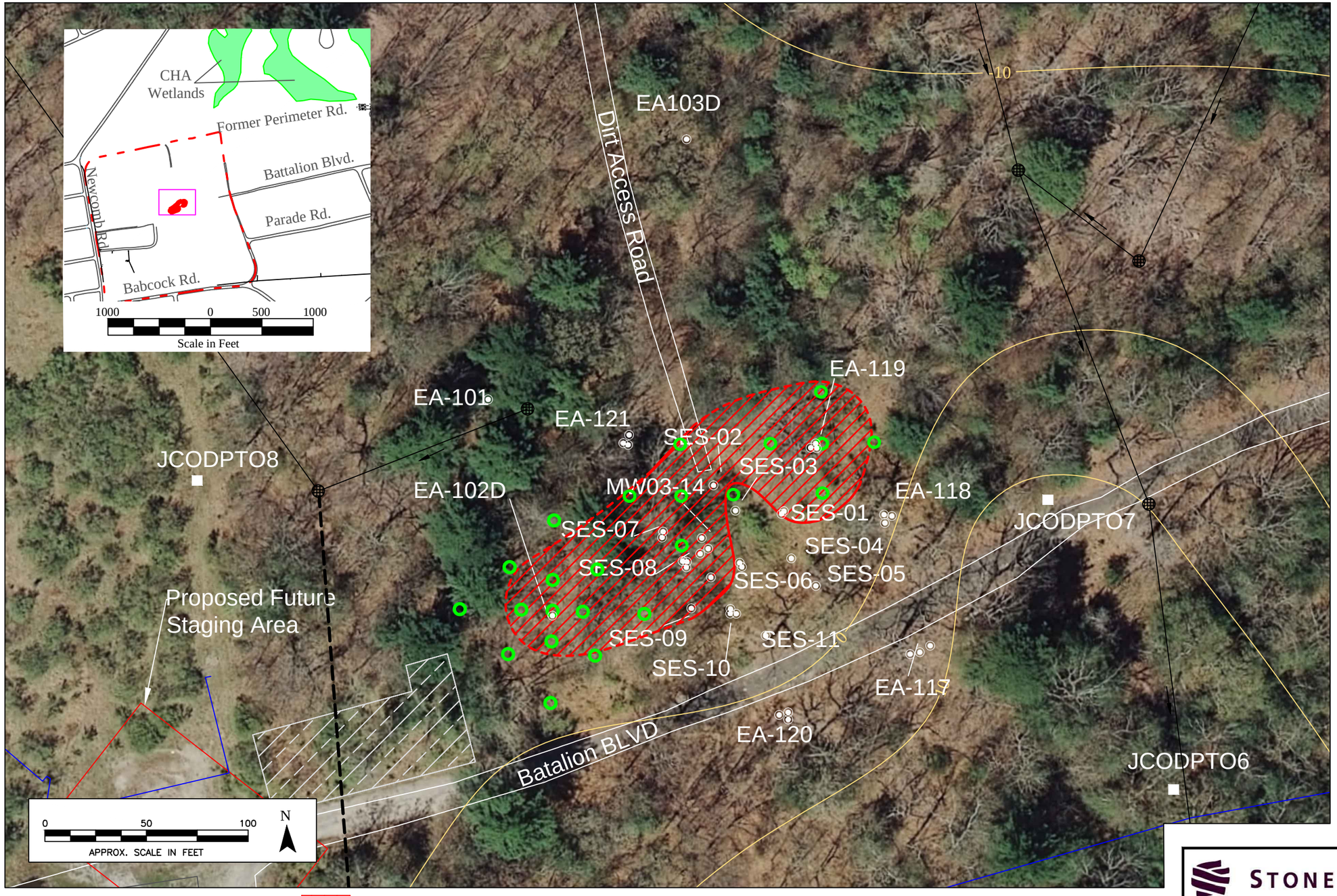


O:\PROJ-10\R2305-R USACE\NAE HTRW SB CONTRACT\05_USACE NIKE PR-58 FS N. KINGSTOWN RI\E-DATA\AUTOCAD\soil borings

LEGEND

- Monitoring Well Location
- Groundwater Profiling Location
- Source Area
- Top of Bedrock Elevation Contours
- Proposed Soil Boring
- Drainage Line
- Map Extent
- Historical Concrete Pad/Slab
- Abandon Water Line
- Unknown Drainage Line
- Catch Basin

Well Key:

"S" Wells are screened across water table or above ISC silty-sand/silt/clay unit.

"D" Wells are screened below ISC silty-sand/silt/clay unit and/or within five feet of the bedrock surface.

"R" Wells are open holes or screened in bedrock.

Notes:

1) Approximate source area location delineated using sample data from groundwater monitoring wells (2009 and 2010 sampling events) screened within deep overburden or bedrock that showed PCA and/or TCE concentrations in excess of 1% of their single-component solubility.

2) Historical soil sampling and groundwater profiling data (2008 event) also were considered in determining the approximate source area location.

Orthophotography data collection flown April 29 through May 2, 2011. Rhode Island Geographic Information System(RIGIS, 2011).

Survey and GPS data collection performed by the New England district of the Unitied States Army Corps of Engineers as part of the 2007 synoptic groundwater sampling program.

GPS data collection performed by the New England District of the Untied States Army Corps of Engineers - 11/5/08 and 9/24/09.

Final closure and post-closure plan for Peabody Clean Industries hazardous waste tank farm (era, 1984).

DATUMS

Coord. System: Rhode Island State Plane
Horizontal Datum: feet NAD 1983
Vertical Datum: feet NAVD 1988

REVISIONS

#	Date	Drwn	Chk'd	App'd	Description
1	10/12 /12	DJL	10/12 /12	JS	Updated orthos, sources, staging area, utilities updated
2	3/13 /13	DJL	3/13 /13	JS	Updated concrete pad location
3	4/26 /13	DJL	4/26 /13	JS	Updated soil boring locations

STONE ENVIRONMENTAL INC

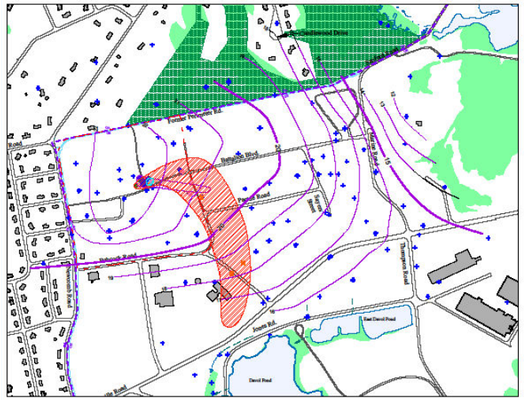
TITLE: Proposed Source Area Soil Boring Locations

PROJECT: Former Nike Battery PR-58

FIGURE NO.: 7

DATE: 4/26/13 DRAWN BY: DJL CHECKED BY: JS

Extent of PCE Contamination in Groundwater



GROUNDWATER STANDARDS/OBJECTIVES

Analyte: Tetrachloroethene (PCE)

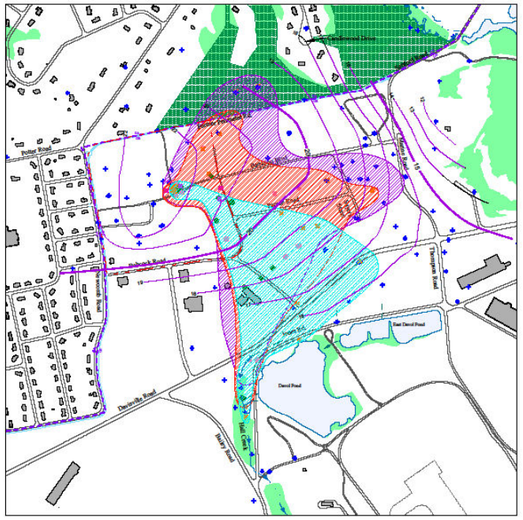
MCL/RI GA GWQO: 5 ppb

RI GB UCL: None Available

LEGEND

- 0 to <5 ppb
- 5 to <10 ppb
- 10 to <100 ppb
- 100 to <1,000 ppb
- 1,000 to <400,000 ppb

Extent of PCA Contamination in Groundwater



GROUNDWATER STANDARDS/OBJECTIVES

Analyte: 1,1,2,2-Tetrachloroethane (PCA)

MCL/RI GA GWQO: None Available

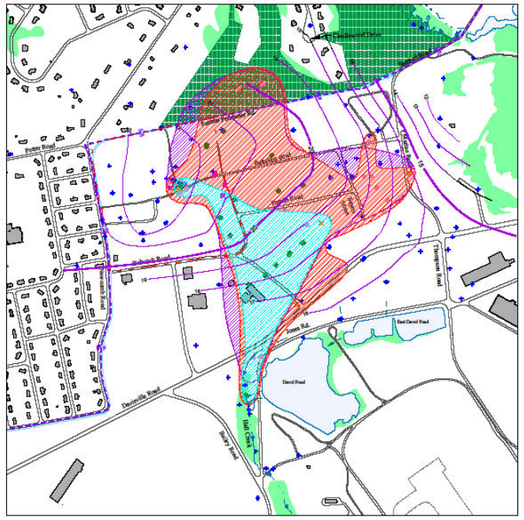
RI GB UCL: None Available

LEGEND

- 0 to <1 ppb
- 1 to <10 ppb
- 10 to <100 ppb
- 100 to <1,000 ppb
- 1,000 to <400,000 ppb

Note: Extent of detections represented where no MCL exists

Extent of TCE Contamination in Groundwater



GROUNDWATER STANDARDS/OBJECTIVES

Analyte: Trichloroethene (TCE)

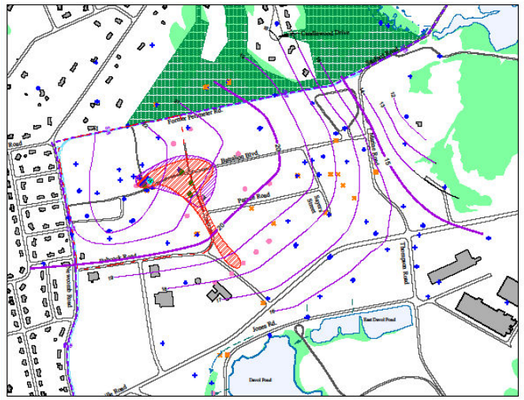
MCL/RI GA GWQO: 5 ppb

RI GB UCL: 87,000 ppb

LEGEND

- 0 to <5 ppb
- 5 to <10 ppb
- 10 to <100 ppb
- 100 to <1,000 ppb
- 1,000 to <400,000 ppb

Extent of *trans*-1,2-DCE Contamination in Groundwater



GROUNDWATER STANDARDS/OBJECTIVES

Analyte: *trans*-1,2-Dichloroethene (*trans*-1,2-DCE)

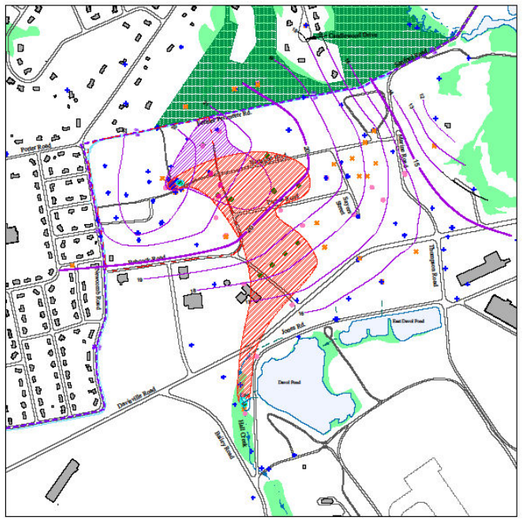
MCL/RI GA GWQO: 100 ppb

RI GB UCL: 79,000 ppb

LEGEND

- 0 to <1 ppb
- 1 to <10 ppb
- 10 to <100 ppb
- 100 to <1,000 ppb
- 1,000 to <400,000 ppb

Extent of *cis*-1,2-DCE Contamination in Groundwater



GROUNDWATER STANDARDS/OBJECTIVES

Analyte: *cis*-1,2-Dichloroethene (DCE)

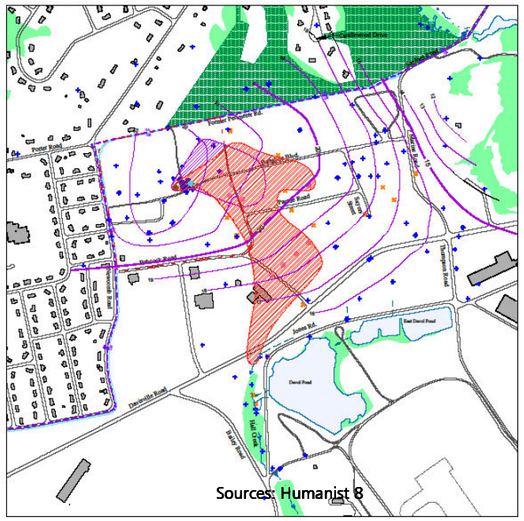
MCL/RI GA GWQO: 70 ppb

RI GB UCL: 69,000 ppb

LEGEND

- 0 to <1 ppb
- 1 to <10 ppb
- 10 to <70 ppb
- 70 to <1,000 ppb
- 1,000 to <400,000 ppb

Extent of 1,1,2-TCA Contamination in Groundwater



GROUNDWATER STANDARDS/OBJECTIVES

Analyte: 1,1,2-Trichloroethane (1,1,2-TCA)

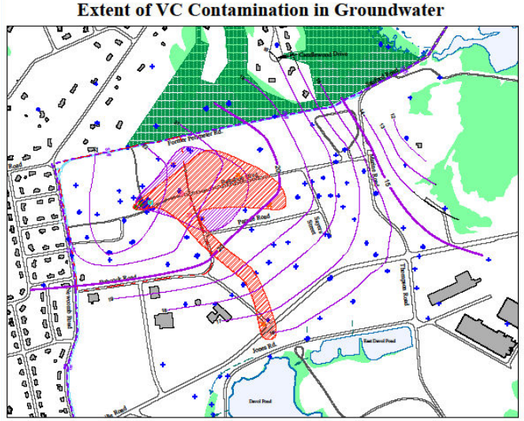
MCL/RI GA GWQO: 5 ppb

RI GB UCL: Not Available

LEGEND

- 0 to <1 ppb
- 1 to <5 ppb
- 5 to <100 ppb
- 100 to <1,000 ppb
- 1,000 to <400,000 ppb

Extent of VC Contamination in Groundwater



GROUNDWATER STANDARDS/OBJECTIVES

Analyte: Vinyl Chloride (VC)

MCL/RI GA GWQO: 2 ppb

RI GB UCL: Not Available

LEGEND

- 0 to <2 ppb
- 2 to <10 ppb
- 10 to <100 ppb
- 100 to <1,000 ppb
- 1,000 to <400,000 ppb

Scale in Feet: 700 0 350 700 1400 2800

SOURCES

Orthophotography and LiDAR Terrain Data Collection, Flown April 7-8, 2007, Rhode Island Formerly Used Defense Sites (Sky, 2008).

Environmental Geographic Information System Release 1.1 by Tetra Tech NUS, (TetraTech NUS, 2008).

Survey and GPS data collection performed by the USACE NAE as part of the 2007 Synoptic Groundwater Sampling Program.

GPS data collection performed by the USACE NAE - 11/05/08 and 09/24/09.

Survey by Environmental Planning and Surveying, Inc. - 12/2008, 11/2009, and 04/2010.

Rules and Regulations for the Investigation and Remediation of Hazardous Material Releases (RIDEM, 2004a).

USEPA Regional Screening Levels (RSLs) (USEPA, 2010).

Sources: Humanist 8

GLOBAL LEGEND

- Former Nike Battery PR-58 Site Project Area
- Rhode Island Department of Environmental Management GA/GB Groundwater Classification Boundary
- Culvert with Flow Direction
- Creek with Flow Direction
- Wetland
- Water Body
- Building
- Extent at or above MCL in Bedrock
- Extent at or above MCL in Deep Overburden Aquifer
- Extent at or above MCL in Shallow Overburden Aquifer
- Bike Path
- Carriage Hill Association Common Land
- 10' Groundwater Contour (Bedrock Aquifer, 10/15/09)
- 2' Groundwater Contour (Bedrock Aquifer, 10/15/09)

NOTES

1) Rhode Island GA/GB Groundwater Quality Objective (RI GA/GB GWQO) Boundary as determined by Rhode Island Department of Environmental Management (RIDEM)
   - -GA indicates area suitable for water supply usage
   - -GB indicates area not suitable for water supply usage
2) Wells located on Carriage Hill Association Common Land not accessible during the 2009-2010 Remedial Investigation
3) MCL-Maximum Contaminant Level as set by the United States Environmental Protection Agency
4) RI GB UCL-RIDEM Upper Concentration Limit

O:\Proj-10\IR2305-R USACE NAE HTRW SB Contract\03_NikePR-58\RIFS\Data\MapDocuments\Report\Fig8_Extent_of_CVOCs_JCo.mxd

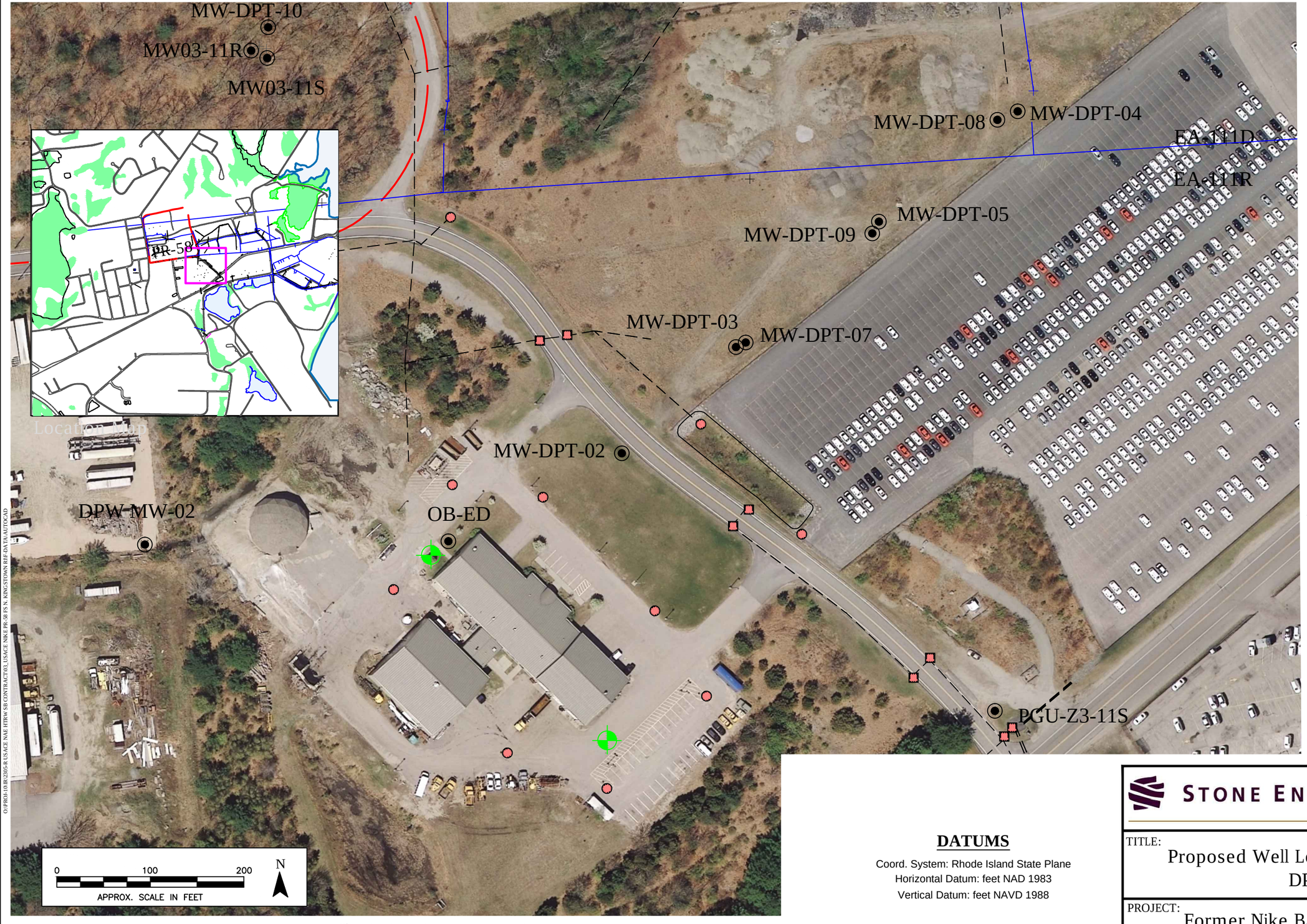
STONE ENVIRONMENTAL INC

Sources: All Figure Data: The Johnson Company, from Draft Remedial Investigation Report. Johnson Company (October 2011) Sources: Orthophotography and LiDAR Terrain Data Collection: Flown April 7-8, 2007, Rhode Island Formerly Used Defense Sites (Sky, 2008); Environmental Geographic Information System Release 1.1, Tetra Tech NUS; Survey and GPS Data Collection: USACE NAE; Survey Data: Environmental Planning and Surveying; Rules and Regulations for the Investigation and Remediation of Hazardous Materials Releases: RIDEM, 2004a; USEPA Regional Screening Levels: USEPA, 2010.

Contaminant Plume Maps

REMEDIAL INVESTIGATION / FEASIBILITY STUDY SUPPLEMENTAL WORK PLAN, NIKE BATTERY SITE PR-58 NORTH KINGSTOWN, RHODE ISLAND

Figure 8



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Note:
Source- orthophotography data collection flown April 29 through May 2, 2011. Rhode Island Geographic Information System(RIGIS, 2011).
Survey and GPS data collection performed by the New England District of the United States Army Corps of Engineers as part of the 2007 Synoptic Groundwater Sampling Program.
-GPS data collection performed by the USACE NAE - 11/05/08 and 09/24/09
-Survey by Environmental Planning and Surveying, Inc. - 12/08, 11/09, and 04/10
Data Provided Electronically by the Quonset Point Development Company, Nike Site.dwg and bikepath.dwg, April 2011 (QDC, 2011)

LEGEND

- PR-58 Property Boundary
- Proposed Monitoring Wells
- Existing Monitoring Wells
- Map Extent
- Round Catch Basin
- Grated Catch Basin
- Drain Line
- Water Line

DATUMS

Coord. System: Rhode Island State Plane
Horizontal Datum: feet NAD 1983
Vertical Datum: feet NAVD 1988

REVISIONS

#	Date	Drwn	Chk'd	App'd	Description
1	10/30/12	DJL	JS	10/30/12	Displayed 2011, DPT wells shown. Orthos, Notes-sources updated, drainage.

STONE ENVIRONMENTAL INC

TITLE: Proposed Well Locations, North Kingstown DPW Property

PROJECT: Former Nike Battery PR-58

FIGURE NO.: 9

DATE: 11/12/12

DRAWN BY: DJL

CHECKED BY: JS

APPENDICES

APPENDIX A: SAMPLING AND ANALYSIS PLAN

SAMPLING AND ANALYSIS PLAN FOR REMEDIAL INVESTIGATION / FEASIBILITY STUDY SUPPLEMENTAL INVESTIGATION

FINAL

FORMER NIKE BATTERY PR-58
NORTH KINGSTOWN, RHODE ISLAND

FUDS Project Number: D01RI0029 05
CENAE Project Number: 108346

Contract Number: W912WJ-11-D-0001
Stone Project ID 102305-R
October 2013

Prepared for:

US Army Corps of Engineers, New England District
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Prepared by:

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STONE ENVIRONMENTAL INC

COMMITMENT TO IMPLEMENT THE ABOVE SAMPLING AND ANALYSIS PLAN		
Joseph Schmidl, PG, CWS	[signature]	4 November 2013
Contractor's Project/Task Manager (print)	Signature	Date
Kim Watson	Kim B. Watson	4 Nov. 2013
Contractor's QC Manager (print)	Signature	Date
Other as Appropriate/Affiliation* (print)	Signature	Date
Other as Appropriate/Affiliation* (print)	Signature	Date
Other as Appropriate/Affiliation* (print)	Signature	Date

* Commitment signature is required for any ancillary sampling, analytical, or data assessment support provided by a contactor or subcontractor. For example, the Contractor's laboratory QC manager or director should sign the title page if analytical services are provided.

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Figure 4: Proposed Well Locations, North Kingstown DPW Property

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Figure 5b: Proposed Monitoring Well Sampling Locations - South

Figure 6: Proposed Residential Water Supply Wells Sample Locations

LIST OF ACRONYMS

AHA	Activity Hazard Analyses
AST	above ground storage tanks
APP	Accident Prevention Plan
ARARs	Applicable or relevant and appropriate requirements
BTOC	below the top of the well casing
bvcA	vinyl chloride reductase
CERCLA	Comprehensive Environmental Response, Compensation and Liability Act
CHA	Carriage Hill Association
cm	centimeters
CoC	chain of custody
COC	contaminants of concern
CSM	conceptual site model
CSS	Contract Compliance Screening
CVOCs	chlorinated volatile organic compounds
DANC	decontamination agent non-corrosive
DCE	dichloroethene
DNAPL	dense non-aqueous phase liquid
DO	dissolved oxygen
DPT	direct-push technology
DPW	Department of Public Works
DQOs	data quality objectives
EDD	electronic data deliverable
EDMS	Environmental Data Management System
eV	electron Volt
fbgs	feet below ground surface
FEP	fluorinated ethyl propylene
FID	flame ionization detector
FOC	fraction organic carbon
FOL	field operations leader
FS	Feasibility Study
FSP	Field Sampling Plan
ft	feet/foot
FUDS	Formerly Used Defense Sites
GC/MS	Gas Chromatography/Mass Spectrometry
GPR	ground-penetrating radar
GPS	global positioning system
GSA	US General Services Agency
HDPE	high-density polyethylene
HHRA	Human Health Risk Assessment
ID	identification
IDW	investigation-derived waste
JCO	The Johnson Company

LCS	Laboratory Control Samples
LOD	Limit of Detection
LOQ	Limit of Quantitation
MCL	maximum contaminant level
mg/L	milligrams per liter
mg/Kg	milligrams per kilogram
MHz	megaHertz
mL	milliliter
MS/MSD	matrix spikes/matrix spike duplicates
mV	milliVolt
NAPL	non-aqueous phase liquid
NAS	Naval Air Station
NAVD	North American Vertical Datum of 1988
NCBC	Naval Construction Battalion Center
NCR	non-conformance report
NELAP	National Environmental Laboratory Accreditation Program
NTU	nephelometric turbidity units
SHE-corrected ORP	standard hydrogen electrode-corrected oxygen-reduction potential
PBC	performance-based contract
PCBs	polychlorinated biphenyls
PCE	tetrachloroethene
PCI	Peabody Clean Industries
PCOCs	potential contaminants of concern
pH	log of hydrogen ion concentration
PID	photoionization detector
PPE	personal protection equipment
ppm v/v	parts per million by volume
PRG	Preliminary Remediation Goal
Property	former Nike Battery PR-58 property
PWS	Performance Work Statement
QAPP	Quality Assurance Project Plan
QA/QC	Quality Assurance/Quality Control
QAO	quality assurance officer
QDC	Quonset Development Corporation
qDHC	Dehalcoccoides spp.
RAO	Remedial Action Objective
RBC	Risk-Based Concentration
RI	Remedial Investigation
RIDEM	Rhode Island Department of Environmental Management
RIEDC	Rhode Island Economic Development Corporation
RSL	regional screening level
SAP	Sampling and Analysis Plan
SEC	Sirrine Environmental Consultants
SEDD	staged electronic data deliverable

SER	Steam Enhanced Recovery
SIM	Selective Ion Monitoring
SLERA	Screening Level Ecological Risk Assessment
SOP	Standard Operating Procedure
SOW	scope of work
SPME	Solid Phase Microextraction
sq. ft	square feet
SSHP	Site Safety and Health
Stone	Stone Environmental, Inc.
TAL	Target Analyte List
TBD	to be determined
TCA	trichloroethane
TCE	trichloroethene
tceA	TCE reductase
TeCA	tetrachloroethane
TIC	tentatively identified compound
TMB	trimethylbenzene
TPH	total petroleum hydrocarbons
µg/Kg	micrograms per Kilogram
USACE	United States Army Corps of Engineers
USCS	Unified Soil Classification System
USEPA	US Environmental Protection Agency
UST	underground storage tank
VC	vinyl chloride
vcrA	vinyl chloride reductase
VOC	volatile organic compound
Work Plan	Feasibility Study Supplemental Work Plan
XML	extensible markup language

1. INTRODUCTION

This Sampling and Analysis Plan (SAP) has been prepared by Stone Environmental, Inc. (Stone) as a component of the *Remedial Investigation / Feasibility Study Supplemental Work Plan* (Work Plan) to support finalization of a Remedial Investigation (RI) and planned Feasibility Study (FS) activities at and near the former Nike Battery PR-58 property (the "Property") in North Kingstown, Rhode Island (Figure 1). Stone will complete this work under a performance-based contract with the United States Army Corps of Engineers (USACE), New England District, which will be completed as part of the USACE Formerly Used Defense Sites (FUDS) Program. The ultimate goal of USACE for this Task Order is to propose a remedy for the Site (a Proposed Plan), and select the final remedy as part of a Decision Document.

1.1. Project Organization

The project is being performed for USACE, New England District. USACE will provide execution and technical oversight as well as administrative support and review. The project is being performed under the regulatory oversight of the Rhode Island Department of Environmental Management (RIDEM). Other stakeholders in the project include the United States Environmental Protection Agency (US EPA), the US Navy for the adjacent Naval Construction Battalion Center (NCBC), and the Quonset Point Development Corporation. Stone will perform the work with the assistance of subcontractors for remedy design (Watermark Environmental, Inc.), risk assessment (AMEC), fixed laboratory analysis (ESS Laboratory and others), subsurface utility location (Hager-Richter Geosciences, Inc.), and surveying (Environmental Planning and Surveying). The details of the project organization structure are provided in Section 10 of the Work Plan.

1.2. Format of Sampling and Analysis Plan

Per Task 4 of the *Performance Work Statement (PWS)*, issued by USACE on November 5, 2010 and subsequently modified on October 31 and December 12, 2011 July 31, 2012, and February 4, 2013, this SAP has been prepared in accordance with the guidelines provided in *Requirements for the Preparation of Sampling and Analysis Plans*(EM 200-1-3), dated February 1, 2001, as well as Table 2-4 and Appendix B of US EPA's *Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA* (EPA/540/G-89/004), dated October 1988. This SAP consists of the following key elements: a Field Sampling Plan (FSP) in Section 2.0, and a Quality Assurance Project Plan (QAPP) in Section 3.0 and Appendix A.

1.2.1. Field Sampling Plan

The FSP has been prepared to include the elements described in Appendix B of US EPA's *Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA* (EPA/540/G-89/004), dated October 1988. The FSP is divided into subsections for each specific method/procedure, wherein each of the FSP elements is described in detail.

1.2.1.1. Site Background

An analysis of existing data is included in Sections 2 and 3 of the Work Plan; for this reason, a brief summary of the existing data is included in the FSP. This analysis includes a description of the Property and surrounding areas and a discussion of known and suspected contaminant sources, probable transport pathways, and other information about the project, found in Work Plan Section 2; the method/procedure-specific background information are briefly reiterated in each subsection. The analysis also includes descriptions of specific data

gaps and ways in which sampling is designed to fill those gaps, found in Work Plan Section 3, and is reiterated in the initial subsection for each field sampling task, in an effort to help orient the sampling team in the field.

1.2.1.2. Sampling Objectives

The specific objectives of each sampling method/procedure that describe the intended uses of data are clearly and succinctly stated in each subsection of the FSP.

1.2.1.3. Sample Location and Frequency

This subsection of each sampling method/procedure described in the FSP identifies each sample matrix to be collected and the constituents to be analyzed. The number of samples to be collected is indicated in each subsection. The appropriate number of replicates and blanks, is clearly identified in Table 5 in Subsection 2.10. A figure is referenced to show the locations of existing or proposed sample locations.

1.2.1.4. Sampling Equipment and Procedures

For each sampling method/procedure, a step-by-step description, cross-referenced to the appropriate Stone Standard Operating Procedure (SOP), has been provided, in order to enable the field team to gather data that will meet the data quality objectives (DQOs). A list of the equipment to be used and its material composition, along with decontamination procedures, is also included.

1.2.1.5. Sample Designation

A master sample numbering system has been established for this project, and is described in subsection 2.9. The sample designation includes the sample or well number, the sampling round, the sample matrix (e.g., surface soil, groundwater, soil boring), and an identifier for the project.

1.2.1.6. Sample Handling and Analysis

The discussion of sample handling and analysis for the RI/FS investigation tasks is included in subsection 2.10. This subsection includes a table that identifies the number and type of samples to be collected, Quality Assurance / Quality Control (QA/QC) sample requirements, sample preservation methods, types of sample containers, shipping requirements, and holding times. This subsection also cross-references applicable portions of the QAPP.

1.2.2. Quality Assurance Project Plan

Per Task 4 of the *Performance Work Statement (PWS)*, issued by USACE on November 5, 2010 and subsequently modified on October 31 and December 12, 2011, the QAPP has been prepared in accordance with *Uniform Federal Policy for Quality Assurance Project Plans* (DTIC ADA 427785), dated March 2005. Standard Operating Procedures are provided in Appendix B and Laboratory SOPs are provided in Appendix C of this document.

1.2.3. Health and Safety

Stone has produced and implemented a written safety and health program meeting the requirements of all applicable federal, state, and local laws and regulations, and approved by USACE's contracting officer. Stone will ensure that its subcontractors, suppliers, and support personnel follow all safety and health provisions established in the approved Accident Prevention Plan (APP), Site Safety and Health Plan (SSHP), and Activity

and Hazard Analyses (AHA), found in Appendix B of the Work Plan. USACE reserves the right to stop work under this contract for any health and safety violations at no additional cost to USACE. Once USACE verifies that corrective action has been implemented, Stone will be able to continue performance under the contract. The SSHP contains the following elements: project description and contaminant characterization, safety and health hazard(s) assessment and risk analysis, organization and responsibilities of safety and health staff, site-specific training and medical surveillance parameters, personal protective equipment (PPE) and decontamination facilities with documented procedures to be used for same, monitoring and sampling requirements, safety and health work precautions and procedures, site control measures, onsite first aid and emergency equipment, emergency response plans and contingency procedures (onsite and offsite), logs, reports, and record keeping.

1.3. Data Gap Analysis

Stone submitted a *Draft Data Gaps Analysis Report* (Stone, 2012) to USACE in May 2012, which identified thirteen data gaps for the Project. These data gaps, which are discussed in detail in Subsection 3.1 of the Work Plan, include the following:

1. Questionable representativeness of dissolved metals data in shallow aquifer ;
2. Lack of pore water, surface water, and sediment data from Hall Creek under "worst case" seasonal low water table conditions;
3. Insufficient Source-Area characterization;
4. Lack of soil treatment parameters in the Source Area;
5. Minimal groundwater quality data from certain wells;
6. Lack of current geochemical data to evaluate natural attenuation;
7. Lack of primary porosity data from the bedrock;
8. Lack of groundwater data characterizing Carriage Hill Association (CHA) wetlands interconnection;
9. Lack of shallow groundwater quality data at the North Kingstown Department of Public Works (DPW) property;
10. Lack of human health risk assessment for areas east and south-southeast of the PR-58 Property;
11. Lack of drinking water quality data for several residential water supply wells;
12. Lack of vapor intrusion exposure pathway data on CHA land north of the PR-58 Property; and
13. Insufficient shallow monitoring well network east of the PR-58 Property.

1.3.1. Remedial Investigation / Feasibility Study Task Development

As stated in Subsection 4.2 of the Work Plan, the RI/FS tasks comprise the list of investigative tasks that are required to achieve the objectives of the RI/FS, namely, to eliminate the identified data gaps. The list of RI/FS tasks has been developed in an iterative fashion, with input and approval from USACE and Stone through the following steps:

1. Initial SOW tasks were included in the *PWS*, dated November 5, 2010;

2. Subsequent discussions between USACE and Stone resulted in revisions to the *PWS*, dated on October 31, 2011, December 12, 2011, March 26, 2012, June 15, 2012, and July 11, 2012;
3. Additional data gaps were identified by Stone under Task 2 of the *PWS*, summarized in the *Draft Data Gaps Analysis Report*, dated May 25, 2012 (Stone, 2012);
4. In a conference call with Stone on August 16, 2012, USACE requested that, in addition to evaluating the possible presence of non-aqueous phase liquid (NAPL) in the vicinity of monitoring well EA-102D, the purpose of the "Additional Source-Area Characterization" task will be to further refine the geometry of the Source Area (primarily to the north and east). Further refinement of the Source-Area geometry will be funded via Optional Task 19 of Mod 01 ("Expanded Source-Area Investigation").
5. In a conference call with Stone on November 7, 2012, USACE requested that, in addition to collecting co-located pore water, surface water, and sediment sample collection from the vicinity of Hall Creek, an additional set of samples be collected from a wetland to the north of the Property on private property at 710 Fletcher Road.

A summary of the data gaps and proposed data collection tasks and methods to eliminate them is provided in Table 1 of the Work Plan. It should be noted that data gap 11 is not being addressed as part of the RI/FS investigation due to property access issues. Data gap 12 is being addressed under a separate mechanism.

1.3.2. Remedial Investigation / Feasibility Study Field Task Summary

Six RI/FS field tasks were developed to address the data gaps identified in Sections 3 and 4 of the Work Plan. Many of the tasks serve to eliminate more than one data gap, and also provide benefits in addition to data gap reduction. Table 1 summarizes the six RI/FS tasks, and provides a breakdown of the sampling methods / procedures required to accomplish each task.

Table 1: Summary of Feasibility Study Field Tasks, Data Quality Objectives, and Sampling Methods

RI / FS Field Task	Data Quality Objective(s)	Sampling Methods/Procedures Required
Collect additional pore water, surface water, and sediment samples from Hall Creek and vicinity	Schedule the sampling during annual low-flow period. Collect all samples according to the approved methodologies identified in the SAP. Document sample locations and depth intervals in accordance with the requirements of the SAP. The LOQs for all the chemical parameters must be less than the screening level ecological risk assessment (SLERA) risk-based screening limits.	Pore water sampling; surface water sampling; sediment sampling, sample location; sample analysis; water level measurement, maintenance and decontamination of field equipment, site restoration.

RI / FS Field Task	Data Quality Objective(s)	Sampling Methods/Procedures Required
Collect additional pore water, surface water, and sediment samples from a wetland north of the Property	Schedule the sampling during annual low-flow period. Collect all samples according to the approved methodologies identified in the SAP. Document sample locations and depth intervals in accordance with the requirements of the SAP. The LOQs for all the chemical parameters must be less than the screening level ecological risk assessment (SLERA) risk-based screening limits.	Pore water sampling; surface water sampling; sediment sampling, sample location; sample analysis; water level measurement, maintenance and decontamination of field equipment, site restoration.
Analysis of existing rock core for primary porosity	Achieve analytical reporting limit objectives, such that porosity data are usable for determination of potential matrix diffusion in the bedrock portion of the aquifer.	Rock core sampling; sample analysis, maintenance and decontamination of field equipment.
Collection and analysis of groundwater samples from approximately 50 monitoring wells	Identify appropriate monitoring wells for sampling. Perform groundwater sampling in accordance with low-flow methodology to obtain representative samples. The LOQs for all the chemical parameters must be less than the project's decision limits, in order to support the following uses: • Provide additional groundwater trend data at individual wells; • Provide current geochemical data regarding natural attenuation; and • Evaluate dissolved metals concentrations in shallow overburden groundwater.	Water level measurement; low-flow groundwater sampling; sample analysis, maintenance and decontamination of field equipment, management of investigation derived waste (IDW).
Collection and analysis of water samples from residential private wells	The LOQs for all the chemical parameters must be less than the project's decision limits, in order to assess potential water receptors.	Residential drinking water sampling; sample location; sample analysis, maintenance and decontamination of field equipment.

RI / FS Field Task	Data Quality Objective(s)	Sampling Methods/Procedures Required
Additional Source Area characterization	Delineate the extent of impacted soils >10 mg/Kg total VOCs and the potential NAPL source in three dimensions. Characterize the chemical and physical properties of the potential NAPL to support updating the conceptual site model. Install NAPL monitoring wells at appropriate screened intervals to collect NAPL samples. The LOQs for all the chemical parameters must be less than the project's decision limits. Collection of representative soil and NAPL, samples.	Utility clearance; soil coring and soil sample collection; monitoring well installation and development; site survey; sample analysis, maintenance and decontamination of field equipment, management of IDW; site restoration.
Shallow groundwater assessment at the North Kingstown DPW facility	Install shallow overburden groundwater monitoring wells in accordance with the specifications included in the SAP. Perform groundwater sampling in accordance with low-flow methodology to obtain representative samples to further assess vapor intrusion pathway. The LOQs for all the chemical parameters must be less than the project's decision limits.	Utility clearance; soil coring and soil sample collection; monitoring well installation and development; site survey; slow-purge groundwater sampling; sample analysis, maintenance and decontamination of field equipment, management of IDW; site restoration.
Synchronous groundwater elevation measurements from approximately 50 monitoring wells	Identify appropriate monitoring wells for sampling. Perform groundwater measurements in accordance with appropriate methodology to obtain representative data.	Water level measurement; maintenance and decontamination of field equipment.
Bedrock description and measurement	Perform bedrock measurements in accordance with appropriate methodology to obtain representative data	Bedrock description and measurement

Details regarding sampling methods are found in Section 2.

The sampling methods/procedures required to perform the RI/FS field tasks are identified in the third column of Table 1, and are listed below in Table 2, along with a reference to their subsection in the FSP.

Table 2: Summary of Feasibility Study Field Task Sampling Methods/Procedures and Field Sampling Plan Location

Feasibility Study Field Task Methods/Procedures	Field Sampling Plan Location
Pore Water Sampling	Subsection 2.1
Surface Water Sampling	Subsection 2.2
Sediment Sampling	Subsection 2.3
Rock Core Sampling	Subsection 2.4
Soil Coring and Soil Sample Collection	Subsection 2.5
Monitoring Well Installation and Development	Subsection 2.6

Feasibility Study Field Task Methods/Procedures	Field Sampling Plan Location
Slow-Purge Monitoring Well Sampling	Subsection 2.7
Residential Water Sampling	Subsection 2.8
Sample Identification	Subsection 2.9
Sample Analysis	Subsection 2.10
Utility Clearance	Subsection 2.11
Site Survey	Subsection 2.12
Water Level Measurement	Subsection 2.13
Maintenance and Decontamination of Field Equipment	Subsection 2.14
Management of Investigation-Derived Waste	Subsection 2.15
Site Restoration	Subsection 2.16
Bedrock Description and Measurement	Subsection 2.17

2. FIELD SAMPLING PLAN

2.1. Pore Water Sampling

2.1.1. Site Background

2.1.1.1. Hall Creek Investigation

Previous sampling of the components of the Hall Creek system (pore water, surface water, and sediment) was conducted following a record precipitation event, which may have resulted in non-representative analytical results. The SLERA included a recommendation that additional samples for chlorinated volatile organic compounds (CVOC) analyses of pore water, surface water, and sediment be collected from Hall Creek during "worst case" seasonal low groundwater conditions to confirm that contaminants of concern (COCs) present only a minimal impact to ecological receptors. Additionally, measurements of the vertical hydraulic gradient should be made at the time of sample collection, due to the atypical hydrological conditions present during the previous sampling event. These data are important to determine CVOC concentrations under seasonal low groundwater conditions for comparison to ecological screening levels, in order to support a re-evaluation of the SLERA.

2.1.1.2. Northern Wetland Investigation

Previous sampling of monitoring wells located on CHA property proved inconclusive regarding the potential for the discharge of contaminated groundwater to the adjoining wetlands, and access has not been granted to perform additional sampling of the wells on the CHA property. However, the property at 710 Fletcher Road extends into the western of the two wetlands north of the Property, and access to that property has been granted for the purposes of sampling. Additionally, measurements of the vertical hydraulic gradient will be made at the time of sample collection to confirm the shallow vertical component of the hydraulic gradient.

2.1.2. Sampling Objectives

The objective of this sampling task is to measure the concentrations of CVOCs in the pore water component of the Hall Creek system and a wetland north of the Property. These data will be used to evaluate whether a potential pathway exists for CVOCs to impact the creek and/or wetland and their ecology, and to support the assessment of remedial alternatives in the FS. In addition, pore water and surface water elevation data will be obtained at the Hall Creek and 710 Fletcher Road locations, via the use of staff gauges. The objective is to collect the samples during seasonally-low groundwater conditions, when groundwater elevations drop below their annual mean values following their spring highs, which are anticipated no earlier than June.

The data obtained from these tasks should be of sufficient quality to allow the project team to make decisions regarding CVOC concentrations relative to the risk-based screening levels for pore water, surface water and sediments. In order to support environmental risk assessment, the samples must be collected from the bioactive zone, 15 to 20 centimeters below the surface water-sediment interface. The screening levels used for these evaluations are based on USEPA Region 3 Freshwater Screening Benchmarks for the pore water.

2.1.3. Sample Location and Frequency

2.1.3.1. Hall Creek Investigation

Twelve co-located pore water, surface water, and sediment samples will be collected along Hall Creek, depicted on Figure 2. The proposed locations are the same locations previously sampled by The Johnson Company (JCO) in 2009. The sample locations will be reacquired using either a global positioning system (GPS) pre-loaded with the sample locations, or sample location markers/landmarks, or a combination of both. Samples will be collected at the most downstream location first, and sequentially in the upstream direction thereafter. The samples will be analyzed for VOCs via USEPA Method 8260B by the fixed laboratory. Six of the samples will also be analyzed for total and dissolved metals in accordance with SW 846 methods. Table 5, included in Subsection 2.10, summarizes the number of samples to be collected, including the required number of replicate and blank samples. Information regarding the laboratory requirements for detection limits, precision, and accuracy of pore water samples are summarized in Table 5 and discussed in subsection 2.10 and the QAPP (Appendix A).

2.1.3.2. Northern Wetland Investigation

A set of co-located pore water, surface water, and sediment samples will be collected from the northern wetland, depicted on Figure 2. The proposed location and the staff gauge will be documented using a survey-grade GPS by a surveyor. The samples will be analyzed for VOCs via USEPA Method 8260B and total and dissolved metals in accordance with SW 846 methods by the fixed laboratory. Table 5, included in Subsection 2.10, summarizes the number of samples to be collected, including the required number of replicate and blank samples. Information regarding the laboratory requirements for detection limits, precision, and accuracy of pore water samples are summarized in Table 5 and discussed in Subsection 2.10 and the QAPP (Appendix A).

2.1.4. Sampling Equipment and Procedures

2.1.4.1. Sampling Equipment

- Henry sampler - 36 inches in length (referred to as PPX36).
- GeoTech Peristaltic Pump
- Water quality meter (YSI Model 699xl) with a flow through cell
- LaMotte Model 2020e Turbidimeter, or equivalent
- Decontamination equipment
- Gloves
- Sampling flags
- Field notebook
- Permanent marking pen
- Cleaning solution
- Deionized water
- Trash bag
- Plastic waste bottle

- Sample containers (see QAPP Worksheet #19 for sample container requirements).

2.1.4.2. Sampling Procedure

The pore water samples will be collected as grab samples following surface water sampling, and prior to sediment sampling at each location, using a peristaltic pump. The following parameters will be measured in the field on a grab sample from the sample location using hand-held meters and recorded on field forms or in the field book: turbidity, dissolved oxygen (DO), negative log of hydrogen ion concentration (pH), specific conductance, temperature, and standard hydrogen electrode (SHE)-corrected oxidation-reduction potential (ORP).

1. Pore water samples will be collected according to Stone SOP 6.40.0.
2. Calibrate water quality instruments in accordance with Stone SOPs SEI-5.26.0, SEI-5.28.0, and SEI-5.30.0. SHE-correction will be implemented during calibration of the ORP sensor, by calibrating the instrument using the YSI reference electrode value (i.e., 428 milliVolts (mV)) at the calibration prompt instead of the Zobell solution value of (i.e., 228 mV).
3. Insert the guard rod into the mini-piezometer body so that the guard rod and handle are squeezed together.
4. Push/drive the tip of the Henry Sampler to approximately 20 centimeters (cm) below the sediment/water interface, using a gentle, twisting motion.
5. When the desired depth or refusal is reached, remove the guard rod from the body without disturbing the position of the deployed sampler.
6. Insert a 3/16-inch wooden dowel into the sampler, until it reaches the water. Make a reference mark on the dowel at the top of the Henry sampler. Remove the dowel, and measure the length of dry dowel to determine the depth to water inside the Henry sampler. Using a rule or tape measure, measure the distance between the top of the Henry sampler and the surface water outside the sampler. Record the difference between the water level inside the Henry sampler and the surface water at the sample location, to document the vertical component of the hydraulic gradient.
7. Connect the sample tubing of the sample port to the peristaltic pump, with initial pump discharge directed to the water. As soon as initial turbidity flushes through, connect the discharge to the water quality indicator flow through cell.
8. Activate the peristaltic pump to extract the pore water at a low flow rate, 50 to 200 mL/minute.
9. Monitor turbidity, dissolved oxygen (DO), pH, specific conductance, temperature, and SHE-corrected ORP for stabilization at 2-minute intervals. Record the values on field forms or in the field book. Stabilization is achieved when three consecutive readings are within the following limits:
    - Turbidity ± 5 nephelometric turbidity units (NTU) for readings >5 NTU, or values <5 NTU
    - DO (± 10% for values greater than 2 milligrams per liter (mg/L); 0.5 mg/L for values less than 2 mg/L)

- Specific conductance ± 3%
- Temperature ± 3%
- pH (± 0.1)
- SHE-corrected ORP (± 10 mV)

10. When the six parameters stabilize, record the final values and disconnect the flow through cell. If an upward gradient is observed, it will be noted in the field notes.
11. Collect the sample from a zero headspace vial holder located on the suction side of the pump directly into the sample container. For samples collected for volatile analysis, use pre-preserved 40-milliliter (mL) glass vials with Teflon septa, ensuring no headspace in the closed sample container.

2.2. Surface Water Sampling

2.2.1. Site Background

2.2.1.1. Hall Creek Investigation

Previous sampling of the components of the Hall Creek system (pore water, surface water, and sediment) was conducted following a record precipitation event, which may have resulted in non-representative analytical results. The SLERA included a recommendation that additional samples for CVOC analyses of pore water, surface water, and sediment be collected from Hall Creek during "worst case" seasonal low groundwater conditions to confirm that COCs present only a minimal impact to ecological receptors. Additionally, measurements of the vertical component of the hydraulic gradient should be made at the time of sample collection, due to the atypical hydrological conditions present during the previous sampling event. These data are important to determine CVOC concentrations under seasonal low groundwater conditions for comparison to ecological screening levels, in order to support a re-evaluation of the SLERA.

2.2.1.2. Northern Wetland Investigation

Previous sampling of monitoring wells located on CHA property proved inconclusive regarding the potential for the discharge of contaminated groundwater to the adjoining wetlands, and access has not been granted to perform additional sampling of the wells on the CHA property. However, the property at 710 Fletcher Road extends into the western of the two wetlands north of the Property, and access to this property has been granted for the purposes of sampling. Additionally, measurements of the vertical component of the hydraulic gradient will be made at the time of sample collection to confirm the direction of that gradient in shallow groundwater.

2.2.2. Sampling Objectives

The objective of this sampling procedure is to measure the concentrations of CVOCs in the surface water component of the Hall Creek system and a wetland north of the Property. These data will be used to evaluate whether a potential pathway exists for CVOCs to impact the creek and/or wetland and their ecology, and to support the assessment of remedial alternatives in the FS. In addition, groundwater and surface water elevation data will be obtained at the Hall Creek and 710 Fletcher Road locations, via the use of staff gauges. The objective is to collect the samples during seasonally-low groundwater conditions, when groundwater

elevations drop below their annual mean values following their spring highs, which are anticipated no earlier than June.

The data obtained from these tasks should be of sufficient quality to allow the project team to make decisions regarding CVOC concentrations relative to the risk-based screening levels for surface water. The screening levels used for this evaluation are based on US EPA Region 3 Freshwater Screening Benchmarks for the surface water.

2.2.3. Sample Location and Frequency

2.2.3.1. Hall Creek Investigation

Twelve co-located pore water, surface water, and sediment samples will be collected along Hall Creek, depicted on Figure 2. The proposed locations are the same locations previously sampled by JCO in 2009. The sample locations will be reacquired using either a global positioning system pre-loaded with the sample locations, or sample location markers/landmarks, or a combination of both. Samples will be collected at the most downstream location first, and sequentially in an upstream direction thereafter. The samples will be analyzed for VOCs via US EPA Method 8260B and hardness via US EPA Method 6010 by the fixed laboratory. Six of the samples will also be analyzed for total and dissolved metals in accordance with SW 846 methods. Table 5, included in Subsection 2.10, summarizes the number of samples to be collected, including the required number of replicate and blank samples. Information regarding the laboratory requirements for detection limits, precision, and accuracy of surface water samples are summarized in the QAPP (Appendix A).

2.2.3.2. Northern Wetland Investigation

A set of co-located pore water, surface water, and sediment samples will be collected from the northern wetland, depicted on Figure 2. The proposed location will be documented using a GPS, and the staff gauge will be more precisely located by a surveyor. The samples will be analyzed for VOCs via USEPA Method 8260B and hardness via US EPA Method 6010 and total and dissolved metals in accordance with SW 846 methods by the fixed laboratory. Table 5, included in Subsection 2.10, summarizes the number of samples to be collected, including the required number of replicate and blank samples. Information regarding the laboratory requirements for detection limits, precision, and accuracy of surface water samples are summarized in Table 5 and discussed in Subsection 2.10 and the QAPP (Appendix A).

2.2.4. Sampling Equipment and Procedures

2.2.4.1. Sampling Equipment

- Disposable polyethylene bailer
- Sample tubing
- Peristaltic pump
- Battery pack
- Water quality meter (YSI Model 699xl) with a flow through cell
- LaMotte Model 2020e Turbidimeter, or equivalent
- Sample containers (see QAPP Worksheet #19 for sample container requirements).

2.2.4.2. Sampling Procedure

The surface water samples will be collected as grab samples prior to pore water and sediment sampling at each location, using a disposable bailer or a new glass jar lowered to mid-water. A peristaltic pump may be used as an alternative sampling method if water depths are insufficient. Water depth at the sample location will be recorded in the field book. The following parameters will be measured in the field on a grab sample from the sample location using hand-held meters and recorded on field forms or in the field book: turbidity, DO, pH, specific conductance, temperature, and SHE-corrected ORP.

1. Surface water samples will be collected according to Stone SOP SEI-6.3.4 Surface Water Sampling.
2. Calibrate water quality instruments in accordance with Stone SOPs SEI-5.26.0, SEI-5.28.0, and SEI-5.30.0. SHE-correction will be implemented during calibration of the ORP sensor, by calibrating the instrument using the YSI reference electrode value (i.e., 428 mV) at the calibration prompt instead of the Zobell solution value of (i.e., 228 mV).
3. Measure surface water elevation at nearby staff gauges to the nearest 0.01 ft, as appropriate.
4. Surface water samples will be collected from the most downstream location first, proceeding to upstream locations.
5. The surface water sample will be collected using a disposable bailer or a new glass jar lowered to mid-water. Transfer the sample from the device directly into the sample container. For samples collected for volatile analysis, use pre-preserved 40 mL glass vials with Teflon septa, ensuring no headspace in the closed sample container.
6. A peristaltic pump may be used to collect the sample if water depth is insufficient to immerse a bailer or sample jar. Connect the sample tubing to the peristaltic pump, and activate the peristaltic pump to collect the surface water.
7. Collect the sample from a zero headspace vial holder located on the suction side of the pump directly into the sample container. For samples collected for volatile analysis, use pre-preserved 40-mL glass vials with Teflon septa, ensuring no headspace in the closed sample container.
8. Record temperature, turbidity, DO, pH, specific conductance, and SHE-corrected ORP following sample collection with the sonde of the water quality meter. Record the values on field forms or in the field book. Stabilization is achieved when three consecutive readings are within the following limits:
    - Turbidity ± 5 NTU for readings >5 NTU, or values <5 NTU
    - DO (± 10% for values greater than 2 milligrams per liter (mg/L); 0.5 mg/L for values less than 2 mg/L)
    - Specific conductance ± 3%
    - Temperature ± 3%
    - pH (± 0.1)
    - SHE-corrected ORP (± 10 mV)

2.3. Sediment Sampling

2.3.1. Site Background

2.3.1.1. Hall Creek Investigation

Previous sampling of the components of the Hall Creek system (pore water, surface water, and sediment) was conducted following a record precipitation event, which may have resulted in non-representative analytical results. The SLERA included a recommendation that additional samples for CVOC analyses of pore water, surface water, and sediment be collected from Hall Creek during "worst case" seasonal low groundwater conditions to confirm that COCs present only a minimal impact to ecological receptors. Additionally, measurements of the vertical component of the hydraulic gradient should be made at the time of sample collection, due to the atypical hydrological conditions present during the previous sampling event. These data are important to determine CVOC concentrations under seasonal low groundwater conditions for comparison to ecological screening levels, in order to support a re-evaluation of the SLERA.

2.3.1.2. Northern Wetland Investigation

Previous sampling of monitoring wells located on CHA property proved inconclusive regarding the potential for the discharge of contaminated groundwater to the adjoining wetlands, and access has not been granted to perform additional sampling of the wells on the CHA property. However, the property at 710 Fletcher Road extend into the western of the two wetlands north of the Property, and access to this property has been granted for the purposes of sampling.

2.3.2. Sampling Objectives

The objective of this sampling procedure is to measure the concentrations of CVOCs in the sediment component of the Hall Creek system and a wetland north of the Property. These data will be used to evaluate whether a potential pathway exists for CVOCs to impact the creek and/or wetland, and their ecology, and to support the assessment of remedial alternatives in the FS. The objective is to collect the samples during seasonally-low groundwater conditions, when groundwater elevations drop below their annual mean values following their spring highs, which are anticipated no earlier than June.

The data obtained from these tasks should be of sufficient quality to allow the project team to make decisions as regarding CVOC concentrations relative to the risk-based screening levels for sediments. In order to support environmental risk assessment, the samples must be collected from the bioactive zone, 15 to 20 centimeters below the surface water-sediment interface. The screening levels used for this evaluation are based on US EPA Region 3 Risk-Based Concentrations (RBCs) and Freshwater Sediment Screening Benchmarks for the sediment results.

2.3.3. Sample Location and Frequency

2.3.3.1. Hall Creek Investigation

Twelve co-located pore water, surface water and sediment samples will be collected along Hall Creek, depicted on Figure 2. The proposed locations are the same locations previously sampled by JCO in 2009. The samples are to be collected from the "bioactive" zone, approximately 15 to 20 cm below the sediment-water interface. The sample locations will be reacquired using either a global positioning system pre-loaded with the

sample locations, or sample location markers/landmarks, or a combination of both. Samples will be collected at the most downstream location first, and sequentially in the upstream direction thereafter. The samples will be analyzed for VOCs via USEPA Method 8260B and total organic carbon via the Lloyd Kahn Method by the fixed laboratory. Table 5, included in Subsection 2.10, summarizes the number of samples to be collected, including the required number of replicate and blank samples. Information regarding the laboratory requirements for detection limits, precision, and accuracy of sediment samples are summarized in Table 5 and discussed in Subsection 2.10 and the QAPP (Appendix A).

2.3.3.2. Northern Wetland Investigation

A set of co-located pore water, surface water, and sediment samples will be collected from the northern wetland, depicted on Figure 2. The proposed location will be documented using a GPS. The samples will be analyzed for VOCs via USEPA Method 8260B and total organic carbon via the Lloyd Kahn Method by the fixed laboratory. Table 5, included in Subsection 2.10, summarizes the number of samples to be collected, including the required number of replicate and blank samples. Information regarding the laboratory requirements for detection limits, precision, and accuracy of sediment samples are discussed in Subsection 2.10 and the QAPP (Appendix A).

2.3.4. Sampling Equipment and Procedures

2.3.4.1. Sampling Equipment

- Staff-mounted Ekman Sampler
- Stainless steel trowel
- Sample containers (see QAPP Worksheet #19 for sample container requirements).

2.3.4.2. Sampling Procedure

The sediment samples will be collected as grab samples following surface water and pore water sampling at each location, using an Ekman Sampler.

1. Sediment samples will be collected according to Stone SOP 6.39.0.
2. Select sample locations in areas of fine grained materials (low-energy depositional) rather than coarse grained gravel and pebbles.
3. Open the valves of the Ekman Sampler. Ensure that the two release wires are securely placed around the release pins.
4. Advance the Ekman Sampler to approximately 15 cm below the sediment-water interface. When in position, trigger the sampler valves to close.
5. Withdraw the Ekman Sampler slowly.
6. If sampling with the Ekman Sampler is not feasible due to root mass, collect samples using a stainless steel trowel.
7. Remove large pieces of organic material and live plants from the sample by hand. Finer organic material that is part of the matrix of the sediments should not be removed.

8. Transfer the sample from the Ekman Sampler directly into the sample container, using a stainless steel trowel. For samples collected for volatile analysis, use methanol pre-preserved 40-mL glass vials with Teflon septa.

2.4. Rock Core Sampling

2.4.1. Site Background

It is possible that dense non-aqueous phase liquids (DNAPL) entered fractures in the bedrock during the release history at the PR-58 Property. The bedrock at the PR-58 Property is described as dark grey-black, contorted phyllites/silty sand turbidites (greywacke), dark grey-black, carbonaceous laminated phyllites, and meta-argillites with pyrite and graphite. Bedrock core logs, outcrops, and borehole optical and acoustical televiewer logs show some beds are preferentially solution-weathered and contain open holes, veins, and vugs. These rocks may contain primary (matrix) porosity on the order of 1% to 10%. This degree of porosity is sufficient to allow for diffusion of a large amount of contaminant mass from the dissolution of the DNAPL in the fractures into the pore spaces in the matrix. If present in the matrix porosity, this contaminant mass represents a long-term source of contamination to groundwater in the bedrock fractures. Additionally, it would be very difficult, if not impractical, to remediate this contaminant mass. No data regarding the porosity of the rock matrix exist for the PR-58 project. These data will be generated from existing rock core from the Property that USACE has stored at Fort Devens, Massachusetts.

2.4.2. Sampling Objectives

The objective of this sampling procedure is to measure the primary porosity of the bedrock. These data will be used to evaluate the potential for matrix diffusion to play a significant role in source persistence and the likely performance of Source- Area remedial measures.

The data obtained from these tasks should be of sufficient quality to allow the project team to make decisions regarding porosity in remedial alternative analyses.

2.4.3. Sample Location and Frequency

Twelve previously obtained rock cores will be sampled to obtain material for analysis. The location from which the rock cores were collected was recorded by the entity that collected them. The samples will be selected in order to bias core selection toward those with evidence of primary porosity. Stone will presume that matrix diffusion in rocks with little or no primary porosity is de minimis. Core selection will also be biased toward locations where relatively high dissolved concentrations have been noted.

The samples submitted to the laboratory will be selected to provide sufficient intact volume to allow the laboratory to extract a 1-inch diameter core approximately 1.75-inches long. The samples will be analyzed for porosity via American Petroleum Institute RP-40 method by the fixed laboratory. Information regarding the laboratory requirements for detection limits, precision, and accuracy of rock samples are summarized in Table 5 and discussed in Subsection 2.10 and the QAPP (Appendix A).

2.4.4. Sampling Equipment and Procedures

2.4.4.1. Sampling Equipment

- Hammer and chisel
- Safety glasses and gloves
- Sampling sheets or field book
- Sample containers (see QAPP Worksheet #19 for sample container requirements).

2.4.4.2. Sampling Procedure

1. Rock samples will be collected according to Stone SOP 6.16.4.
2. Select rock samples from existing cores previously collected from locations in the vicinity of the source area.
3. If appropriately-sized core sections are not available, break the core to the appropriate size using the hammer and chisel.
4. Transfer the sample to the sample container.

2.5. Soil Coring and Analyses

2.5.1. Site Background

2.5.1.1. Source Area Investigation

Personnel from the well drilling crew that installed monitoring well EA-102D informed USACE that they had observed NAPL in the settling tub while advancing casing between 10 and 14 ft below ground surface (bgs). Monitoring well EA-102D is located near the northeast portion of the former concrete pad. No samples were collected from the 10 to 14 foot interval during drilling of the well. The screened interval for EA-102D spans approximately 22 ft bgs to 32 ft bgs (i.e., below the zone of reported NAPL). Therefore, hard data are not available with which to determine whether or not such NAPL is present and if so what the composition of the NAPL is.

In addition, the geometry of the Source Area is not well defined to the north and east of monitoring well EA-119D, limited soil volatile organic compound (VOC) data are available within the "core" of the Source Area to evaluate contaminant mass distribution, and there are no soil oxidant demand data. These additional data would be useful in evaluating Source-Area remedial alternatives during the FS, and will be obtained during the RI/FS field program.

2.5.1.2. North Kingstown DPW Shallow Groundwater Investigation

Four COCs related to the PR-58 Property were detected in soil gas samples collected from beneath the North Kingstown DPW building above risk-based concentrations (RBCs) for sub-slab soil gas in April 2010. Based on data from monitoring wells OB-ED and MW-DPT-2, the contaminant plume from the PR-58 Property flows through the deep unconsolidated deposits and the bedrock under the DPW property. Due to the lack of shallow (i.e., water table) wells in the area of the DPW property, there is uncertainty regarding VOC

concentrations immediately beneath the building and DPW property. Shallow groundwater data are needed to determine if a groundwater-soil vapor-indoor air pathway exists for PR-58 contaminants at the North Kingstown DPW property.

2.5.2. Sampling Objectives

2.5.2.1. Source Area Investigation

The objective of this sampling procedure in the Source Area is to verify the presence or absence of NAPL in soil in the vicinity of EA-102D, and, if present, to identify the physical and chemical properties of NAPL and delineate its horizontal and vertical distribution. If NAPL is present, an additional objective will be to advance soil borings for the installation of overburden NAPL monitoring wells (see subsection 2.6). In addition, the investigation will better delineate the magnitude and extent of Source Area contamination. These data will be used to evaluate the nature and extent of the Source Area, and to support the assessment of remedial alternatives in the FS.

The data obtained from this task should be of sufficient quality to allow the project team to make decisions regarding the nature and extent of NAPL in overburden, to be used in remedial alternative analyses. The screening levels used for this task are three-fold, depending on objective:

- All soil analytical results will be compared against applicable RIDEM Remediation Regulation standards for residential direct exposure;
- Soil samples collected during the Source Area investigation with soil headspace headings greater than 100 milligram per Kilogram (mg/Kg) will be visually inspected for the possible presence of NAPL by using “Oil Red O” dye in accordance with Stone SOP SEI-6.41.0; and
- A concentration of 10 mg/Kg total target CVOCs in soil will be used to estimate the extent of the Source Area and to terminate further investigation of Source Area extent.

2.5.2.2. North Kingstown DPW Shallow Groundwater Investigation

The objective of this sampling procedure on the North Kingstown DPW Property is to characterize the subsurface and advance soil borings for the installation of shallow overburden monitoring wells (see Subsection 2.6).

The data obtained from this task should be of sufficient quality to allow the project team to make decisions regarding the nature and extent of dissolved VOCs in shallow overburden, to be used in human health risk assessment. There are no screening levels for this task.

2.5.3. Sample Location and Frequency

2.5.3.1. Source Area Investigation

To further evaluate the western portion of the Source Area, a minimum of 13 soil core locations will be advanced along two, perpendicular transects with monitoring well EA-102D at the center (intersection) of the transects (Figure 3). One soil core will be advanced immediately adjacent to monitoring well EA-102D; the balance of the borings will be spaced 15-feet apart along each transect. The coring program will be expanded in a gridded pattern (with 15-ft spacing) as needed, based on the near-real time data from the initial two transects, to delineate the horizontal and vertical distribution of NAPL. As such, the sample locations will be

modified or expanded as necessary to achieve the objective of delineating the extent of the Source Area; such critical decisions are subject to the approval of USACE and consultation with RIDEM if available in a timely fashion so as to allow field work to continue, as described in Subsection 10.1 of the Work Plan.

An additional 15 soil coring locations will be advanced within the remainder of the Source Area: five within the "core" of the Source Area, five in the eastern portion of the Source Area in the vicinity of EA-119D, and the remaining five to be determined based on the findings from the first 10 locations (Figure 3). The coring program will be expanded in a gridded pattern, with approximately 25-ft spacing between locations, to delineate the horizontal and vertical distribution of total target CVOCs with concentrations >10 mg/Kg in soil.

Eleven days are budgeted to complete the coring, which should be adequate to advance approximately 30 soil borings to the bedrock surface in the vicinity of the Source Area. As described above, the subsequent sample locations will be modified or expanded as necessary in response to the near-real time results of the initial borings.

Soil borings will be advanced to the bedrock surface, expected to be present between about 25 to 35 feet bgs, or until refusal is encountered. Soil cores will be collected in continuous 5-foot intervals, and soil samples will be collected from the top 0- to 1-ft depth interval, and then 2-foot intervals, measured from ground surface. Based on this sampling strategy, an estimated 17 soil samples will be collected from each soil boring (or where observation suggests that gross contamination may be present), which will result in the collection of approximately 510 discrete soil samples. Each soil sample will be field-screened for soil headspace according to Stone SOP SEI-5.29.0. Each soil sample that has a soil headspace photoionization detector (PID) reading greater than 100 parts per million by volume (ppm v/v) will be field-screened for NAPL using "Oil Red O" dye according to Stone SOP SEI-6-41.0. Each soil sample will be analyzed by the onsite laboratory for target VOCs, via modified US EPA Method 8260C. Aliquots of 10 selected soil samples will be further analyzed by a fixed laboratory for total petroleum hydrocarbons (TPH) via US EPA Method 8100M. Sample selection for TPH analysis will be biased towards soils that exhibit the highest PID readings and or visual/olfactory evidence of contamination within the "smear zone" just above and below the water table. In addition, 10 soil samples (five from the sand/gravel unit and five from the basal till unit) will be collected for analysis for the following physical properties: bulk density and porosity by ASTM D7263, and fraction organic carbon (FOC) by ASTM D2974. The FOC samples will be selected from the sample locations where total target VOC concentrations are non-detect (based on soil analytical results), to reduce the potential interferences. Information regarding the laboratory requirements for detection limits, precision, and accuracy of soil samples are summarized in Table 5 and further discussed in Subsection 2.10 and the QAPP (Appendix A).

2.5.3.2. North Kingstown DPW Shallow Groundwater Investigation

The proposed soil boring locations for two shallow overburden groundwater monitoring wells to be installed at the North Kingstown DPW property are depicted on Figure 4. The soil borings will be advanced to approximately 8 ft below the unconfined shallow groundwater table. In addition to field screening of soil headspace according to Stone SOP SEI-5.29.0, the soil sample from each soil boring with the highest soil headspace reading (or, if no soil headspace readings are elevated, the depth interval immediately above the water table) will be analyzed by the onsite laboratory for target VOCs, via modified US EPA Method 8260C.

2.5.4. Sampling Equipment and Procedures

2.5.4.1. Sampling Equipment

- Geoprobe® direct-push rig (e.g., 6610 DT, 7820DT)
- DT22 dual-tube sampling tool (2.25-inch diameter) or MC5 system used in Piston mode
- PID with an 11.7 electron Volt (eV) lamp
- Appropriate soil sample containers
  - 40 mL VOA vials pre-preserved with methanol.
  - Trimmed HDPE syringes.
  - Clear 125-mL jars for NAPL screening.
- Oil "Red-O" dye.
- Stainless steel scoops and spatulas.
- Electronic balance.
- Field notebook, sample collection form, or other acceptable medium for recording field notes. Ballpoint, indelible pens.
- Shipping materials (coolers, ice, ziplock bags, etc.).
- Sample containers (see QAPP Worksheet #19 for sample container requirements).
- Sample Labels.
- Chain of Custody forms.
- Measuring tape.
- As necessary or as described in the APP/SSHP/AHA appropriate personal protection equipment (PPE) such as gloves.
- Rubber mallet and/or other appropriate hand tools.
- Plastic sheeting for laying out soil samples for characterization.

2.5.4.2. Sampling Procedure

1. Soil cores will be collected utilizing direct-push technology (DPT) per Stone SOP SEI-6.1.6.
2. Pre-clear all drilling locations by calling DigSafe (888-DIG-SAFE) for subsurface utility clearance at least 72 hours prior to drilling activities followed by coordinating directly with QDC and the Town of North Kingstown DPW. In addition, a subsurface utility location contractor will be used to clear the soil boring locations (see Subsection 2.11 for details).
3. The DT22 dual-tube sampling tool and/or MC5 tool will be advanced though each 5-ft interval and retrieved.
4. Following sample description and field screening, an analytical sample will be collected from each 2-ft depth interval. Sample aliquots to be analyzed for VOCs from the undisturbed soil core, without homogenization, into appropriate sample containers. For samples collected for volatile

analysis, use pre-preserved 40-mL glass vials with Teflon septa. For sample aliquots collected for bulk density and porosity, a 5-inch section of undisturbed Macrosampler® liner will be cut and sealed as a sample container.

5. An experienced geologist or other qualified geoscience professional will log the soils as per Stone SOP SEI-6.46.0, using the Unified Soil Classification System (USCS), noting the following characteristics:
    - Particle size distribution
    - Gradation
    - Plasticity
    - Dry strength
    - Color
    - Odor
    - Soil structure
    - Moisture content
    - Consistency
    - Cementation
6. Inspect soil samples visually for the possible presence of NAPL by using "Oil Red O" dye in accordance to standard operating procedure Stone SOP SEI-6.41.0.
7. Screen soil samples for VOCs with a PID in accordance with Stone SOP SEI-5.29.0. The PID will be calibrated daily to 100 ppm v/v isobutylene span gas and to zero using ambient air and a carbon filter.
8. Screen any soil sample that has a soil headspace greater than 100 ppm v/v for NAPL using "Oil Red O" dye according to Stone SOP SEI-6-41.0
9. The sample description, and NAPL and soil headspace screening results will be recorded in a field logbook.
10. Excess sample material will be collected into appropriate storage containers, pending IDW determination.

2.6. Monitoring Well Installation and Development

2.6.1. Site Background

2.6.1.1. Source Area Investigation

Recently, personnel from the well drilling crew that installed monitoring well EA-102D informed USACE that they had observed NAPL in the settling tub while advancing casing between 10 and 14 ft bgs. Monitoring well EA-102D is located near the northeast portion of the former concrete pad. No samples were collected from the 10-14 foot interval during drilling of the well. The screened interval for EA-102D spans approximately 22 ft

bgs to 32 ft bgs (i.e., below the zone of reported NAPL). Therefore, hard data are not available with which to determine whether or not such NAPL is present and if so what the composition of the NAPL is.

2.6.1.2. North Kingstown DPW Shallow Groundwater Investigation

Four COCs related to the PR-58 Property were detected beneath the North Kingstown DPW Building above RBCs for sub-slab soil gas in April 2010. Based on data from monitoring wells OB-ED and MW-DPT-2, the contaminant plume from the PR-58 Property flows through the deep unconsolidated deposits and the bedrock under the DPW property. Due to the lack of shallow (i.e., water table) wells in the area of the DPW property, there is uncertainty regarding VOC concentrations immediately beneath the building and DPW property. Shallow groundwater data are needed to determine if a groundwater-soil vapor-indoor air pathway exists for PR-58 contaminants at the North Kingstown DPW Property.

2.6.2. Sampling Objectives

The objective of this procedure is to install additional overburden monitoring wells to provide additional locations to collect representative groundwater and/or NAPL samples.

The wells installed under these tasks should meet industry standards, and allow the collection of representative groundwater and/or NAPL samples. The standard used for this procedure is based on ASTM D5092.

2.6.3. Sample Location and Frequency

2.6.3.1. Source Area Investigation

Up to three NAPL monitoring wells will be installed in the vicinity of well EA-102D, should mobile NAPL be detected during the Source Area soil investigation described in Subsection 2.5. The proposed locations are unknown at this time, and will be based on the results of the Source Area soil investigation; their locations and screened intervals are considered critical decisions, subject to the approval of USACE and input from RIDEM, as described in Subsection 10.1 of the Work Plan. The general location of these wells is in the western portion of the Source Area, which is depicted in Figure 3. The wells will be designated SEI-03 through -05, sequentially. Information regarding the laboratory requirements for soil samples regarding detection limits, precision and accuracy are summarized in Table 5 and discussed in Subsection 2.10 and the QAPP (Appendix A).

2.6.3.2. North Kingstown DPW Shallow Groundwater Investigation

Two shallow overburden groundwater monitoring wells will be installed at the North Kingstown DPW property, in the vicinity of the North Kingstown DPW Building, as depicted on Figure 4. The wells will be designated SEI-01 and -02, sequentially. If practical, the work at the North Kingstown DPW will be performed prior to work at the Source Area, due to the presumed lower level of contamination. Due to seasonal groundwater fluctuations of up to 9 ft, the screened interval of the groundwater monitoring wells to be installed at the North Kingstown DPW property will be 15 ft, set to straddle the top of the unconfined shallow groundwater table, with 3 ft above and 12 ft below the groundwater elevation observed in the borehole at the time of the investigation, which will be following the seasonal high. This procedure includes only field screening of soil headspace.

Each monitoring well location will be surveyed for location and elevation following installation (see Subsection 2.12 for details of Site Survey methods/procedures).

2.6.4. Sampling Equipment and Procedures

2.6.4.1. Sampling Equipment

- Geoprobe® direct push rig (e.g., 6610 DT or 7820 DT) or equivalent
- Stainless steel trowel
- 2-inch diameter stainless steel well screen and riser (screen slot size to be determined based on soil data) for NAPL wells
- 2-inch diameter, Schedule 40, threaded, polyvinyl chloride (PVC) well screen and riser (screen slot size to be determined based on soil data) for DPW wells
- Silica sand well filter pack (sand size to be determined based on soil data)
- Bentonite chips or pellets
- Bentonite powder
- Cement
- Protective casing
- YSI 699xl water quality meter, or equivalent.
- LaMotte Model 2020e Turbidimeter or equivalent.
- Whale pump and tubing
- Surge block and line
- 5-gallon pail
- Well Construction Log form

2.6.4.2. Sampling Procedure

2.6.4.2.1. Well Installation

11. Monitoring wells will be installed according to SOP SEI-6.4.5.
12. Pre-clear all drilling locations by calling DigSafe (888-DIG-SAFE) for subsurface utility clearance at least 72 hours prior to drilling activities following which Stone will coordinate with QDC and Town of North Kingstown DPW to ensure clearance of the locations. In addition, a subsurface utility location contractor will be used to clear the soil boring locations (see Subsection 2.11 for details).
13. Advance drilling equipment to the desired depth, according to Stone SOP 6.41.0 (see Subsection 2.5).
14. Using soil grain size data obtained from each soil boring, select appropriate sand filter pack and well screen sizes in accordance with ASTM 5092D.
15. Withdraw drill rods and plugs, and set screen/riser in the casing/auger.

16. Groundwater monitoring wells will be constructed of 2-inch diameter, Schedule 40, threaded, PVC well screen and riser, with 15 ft screens. No glue shall be used in the assembly of the well screen/riser.
17. NAPL monitoring wells will be constructed of 2-inch diameter, Schedule 40, threaded, stainless steel well screen and riser, and will include a 1-ft sump below the bottom of the screen interval.
18. Pour filter sand into the annulus between the borehole wall and well screen/riser, to a depth 1 ft above the top of the well screen. Add sand approximately 1 ft at a time, and withdraw casing/augers to match, to prevent binding the well screen/riser in the casing/auger. Account for the greater volume of the borehole after casing/auger removal by withdrawing less casing/auger length than depth of sand added, to maintain a consistent sand filter pack (e.g., do not raise bottom of casing/auger above the level of sand). The movement of sand into the sand pack should be monitored using a weighted tape measure; as the casing/auger is withdrawn, the level of sand in the casing/auger should drop.
19. Pour bentonite chips and/or pellets to the top of the filter pack, to a depth 3 ft above the top of the well screen (2-ft thick layer). If not emplaced below the water table, hydrate the chips/pellets using potable water.
20. Tremie (below the water table) / pump (above the water table) cement/bentonite grout into the annulus between the borehole wall and well riser below the water table, to a depth of 2 ft below ground surface. The grout mix should be 6 gallons of water per 94-lb bag of Type I Portland cement, with no more than 3 to 8% by dry weight bentonite powder.
21. Depending upon the well location and its immediate surroundings, install either a flush-mount waterproof well cover, or a steel protective casing with 2- to 3-ft stick up above ground surface.
22. A 1-foot by 1- foot by 8-inch thick concrete pad level with the ground surface will be placed at each well completed with a well guard. Wire mesh will be incorporated into the concrete pad for added strength. The concrete pad will be beveled at a minimum slope of 1 inch from the casing to the pad perimeter. Well completion will include a lockable well cap.
23. Mark a measurement point clearly with a permanent mark or notch on the top of the well riser (not the well protective casing) for surveying and water level measurement
24. Secure the monitoring well with a locking interior or exterior cap.
25. Prime and paint well protective casings using exterior-grade primer and paint. The well number will be painted on the outside of the guard in block characters. The locking well cap will also be labeled (inside and outside) with the well number and depth using a permanent marker such as a paint pen. Where possible, wells installed in vulnerable locations will be further protected with 2 to 3 steel and concrete bollards to protect them from traffic, snow plows, etc.

2.6.4.2.2. Well Development

1. Allow at least 24 hours following well installation before well development, to prevent mobilization of grout, etc. prior to its setting up.
2. Calibrate water quality instruments in accordance with Stone SOPs SEI-5.26.0, SEI-5.28.0, and SEI-5.30.0.

3. Develop each new well by pumping and mechanical surging, for a minimum of three well volumes. Use development pumping rates sufficient to stress the well and to reduce potential damage to the near-bore aquifer resulting from drilling. The use of acids, dispersing agents or explosives is prohibited.
4. Screen development purge water with a PID, and containerize the purge water.
5. Begin development until development water appears clear to the naked eye.
6. Begin monitoring well development at 5-minute intervals using a water quality meter with a flow through cell until pH, specific conductance and turbidity are stable for three successive measurements. Stability of parameters is defined as follows:
    - +/- 0.2 pH units
    - <10% change in specific conductance
    - <+/- 10% difference in turbidity between two consecutive readings or values <5 NTU.
7. Record the well development procedures and results (e.g., pumping rates, water level drawdown and recovery rates, air monitoring readings, and color of discharged water) on the Well Development Form.

2.7. Monitoring Well Sampling

2.7.1. Site Background

2.7.1.1. Source Area Investigation

Personnel from the well drilling crew that installed monitoring well EA-102D informed USACE that they had observed NAPL in the settling tub while advancing casing between 10 and 14 ft bgs. Monitoring well EA-102D is located near the northeast portion of the former concrete pad. No samples were collected from the 10-14 foot interval during drilling of the well. The screened interval for EA-102D spans approximately 22 feet below ground surface (fbgs) to 32 fbgs (i.e., below the zone of reported NAPL). Therefore, hard data are not available with which to determine whether or not such NAPL is present and if so what the composition of the NAPL is.

2.7.1.2. Groundwater Investigation

2.7.1.2.1. Metals in Groundwater

Prior samples collected to assess the concentration of metals in shallow groundwater were obtained using bailers, which resulted in samples with relatively high turbidity, which in turn resulted in elevated concentrations for some inorganic compounds above US EPA maximum contaminant levels (MCLs). Since analysis of turbid samples result in data that are not representative of true dissolved metals concentrations, the data available are not reliable for assessing the concentrations of metals in shallow groundwater. In order to properly assess the concentrations of metals in shallow groundwater, shallow monitoring wells need to be sampled using the US EPA Low Stress/Low Flow method, which is designed to collect representative groundwater samples that reflect ambient flow conditions with minimal physical and chemical alterations caused by sampling, and reduce turbidity.

2.7.1.2.2. Additional Data at Existing Wells

Approximately 250 wells have been installed on and around the Property over the course of the environmental investigations for the PR-58 project and neighboring US Navy sites. However, some of these wells have a limited sampling history. For example, the wells in the CHA wetlands have only been sampled twice and the cluster of wells along Candlewood Drive has only been sampled once. With such a limited data set it is not possible to assess trends in contaminant concentrations. More current data are needed to determine these trends and current potential for risk. A round of sampling of selected wells, including those with sparse sampling histories, is needed.

2.7.1.2.3. Natural Attenuation Investigation

Three previous assessments of the nature and role of biodegradation in attenuation of groundwater contamination associated with releases at the PR-58 Property have indicated that biodegradation is occurring and may have potential to be a significant mechanism for limiting groundwater transport of contaminants. The first two of these assessments were conducted 14 and 12 years ago, respectively. The most recent assessment was based only on the presence of degradation products and included data collected between 1996 and 2010. There is a need for current geochemical data accompanying current data regarding contaminant concentrations to allow for an assessment of the degree of biodegradation and the likelihood of degradation acting as a significant means of plume attenuation.

2.7.1.3. North Kingstown DPW Soil Vapor Investigation

Four COCs related to the PR-58 Property were detected beneath the North Kingstown DPW Building above RBCs for sub-slab soil gas in April 2010. Based on data from monitoring wells OB-ED and MW-DPT-2, the contaminant plume from the PR-58 Property flows through the deep unconsolidated deposits and the bedrock under the DPW property. Due to the lack of shallow (i.e., water table) wells in the area of the DPW property, there is uncertainty regarding VOC concentrations immediately beneath the building and DPW property. Shallow groundwater data are needed to determine if a groundwater-soil vapor indoor air pathway exists for PR-58 contaminants at the North Kingstown DPW Property.

2.7.2. Sampling Objectives

The objective of this procedure is to collect representative groundwater and/or NAPL samples.

The samples collected should be representative of local groundwater and/or NAPL. The standard used for the groundwater sampling procedure is USEPA's *Low-Stress (low-flow) Purging and Sampling Procedure for the Collection of Groundwater Samples from Monitoring Wells* (EQASOP-GW-001), dated January 19, 2010. The NAPL sampling procedure is based on Stone SOP SEI-6.47.0.

2.7.3. Sample Location and Frequency

2.7.3.1. Source Area Investigation

Up to three NAPL samples will be collected from wells to be installed in the vicinity of well EA-102D, should mobile NAPL be detected during the Source Area soil investigation described in Subsection 2.5. The proposed locations are unknown at this time, and will be based on the results of the Source Area soil investigation, and their locations and screened intervals are considered critical decisions, subject to the approval of USACE and consultation with RIDEM, as described in Subsection 10.1 of the Work Plan. The general location of these

wells will be in the western portion of the Source Area, which is depicted in Figure 3. The screened intervals of these wells will be based on the results of the Source Area soil investigation. The NAPL samples will be analyzed for VOCs via USEPA Method 8260B; SVOCs via USEPA Method 8270C; TPH via USEPA Method 8100M; Petroleum fingerprint via USEPA Method 8015M; PCBs via EPA Method 8082A; Viscosity via ASTM D445; and Specific gravity via ASTM D1298.

Information regarding laboratory requirements for NAPL samples regarding detection limits, precision and accuracy are summarized in Table 5, found in Subsection 2.10, and discussed in Subsection 2.10 and the QAPP (Appendix A). The NAPL sampling program for these wells will be executed in a single day.

2.7.3.2. Groundwater Investigation

Groundwater samples will be collected from up to 51 groundwater monitoring wells for one or more of the following reasons:

- Obtain representative total and dissolved metals samples from approximately 8 shallow overburden monitoring wells. These samples will be analyzed for total and dissolved Target Analyte List (TAL) metals by USEPA Methods 6010/7470/7000;
- Obtain additional VOC results from approximately 51 monitoring wells to support trend analysis. These samples will be analyzed for VOCs via USEPA Method 8260B;
- Obtain representative total and dissolved metals samples from approximately 18 locations located to the south and east of the Source Area, along the migration pathway to the Hall Creek system, in order to determine whether groundwater metals concentrations are influenced by the dissolved VOC plume; and
- Obtain current geochemical data from up to51 monitoring wells to support the evaluation of natural attenuation in the dissolved groundwater plumes. These samples will be field screened for nitrate/nitrite, sulfate/sulfite, ferrous/ferric iron, and chloride using a Hach field test kit, and analyzed by a fixed lab for VOCs via USEPA Method 8260B, ethane/ethene via Method RSK 175, and TOC via US EPA Method 9060A; an additional aliquot will be collected from 10 monitoring wells for analysis for *Dehalcoccoides* spp. (qDHC) by a proprietary method. In addition, should the preceding qDHC analysis indicate that microbial degradation is occurring; additional microbial analyses for TCE reductase (tceA), vinyl chloride reductase (bvcA), vinyl chloride reductase (vcrA) will be performed for samples from the same wells by proprietary methods in order to confirm the specific biodegradation processes present.

The monitoring wells selected for sampling are listed in Table 3 and depicted on Figures 5a and 5b. The table also indicates wells which have dedicated pumps in place. The sampling program for the 51 wells will be performed over an approximately 5-day period. Information regarding the laboratory requirements for groundwater metals, VOCs, and natural attenuation parameter samples regarding detection limits, precision and accuracy are summarized in Table 5 and discussed in Subsection 2.10 and the QAPP (Appendix A).

Table 3: Monitoring Water Wells Proposed for Sampling

Well ID (Depth)	Sampling Rationale	Sampling Parameters/	Pump Type
BR-A (130 ft)	Single, recent result[1]; Distal on north lobe of plume; perimeter well defining plume extent	VOCs, natural attenuation parameters	Bladder
BR-B (61.5 ft)	Single, recent result[1]; Distal West of Source Area; perimeter well defining plume extent	VOCs, natural attenuation parameters	Bladder
BR-D (82.5 ft)	Single, recent result[1]; Distal on south lobe of plume; perimeter well defining plume extent	VOCs, natural attenuation parameters	Bladder
BR-F (90 ft)	Single, recent result[1]; Medial on south lobe of plume; perimeter well defining plume extent	VOCs, natural attenuation parameters, total and dissolved metals	Bladder
EA-102S	Well couplet	Water level	n/a
EA-102D	Six data points[1]; Proximal to Source Area, specifically former concrete pad, where NAPL may have been observed; Total CVOC concentrations are trending upward	VOCs, natural attenuation parameters, total and dissolved metals, qDHC	Bladder*
EA-103S	Well couplet	Water level	n/a
EA-103D	Well couplet	Water level	n/a
EA-104D	Five data points[1]; Proximal northeast of Source Area; shows hydraulic connection to Source Area; closest wells upgradient of CHA property; Total CVOC concentrations are trending upward	VOCs, natural attenuation parameters, total and dissolved metals, qDHC	Bladder*
EA-104R	Six data points[1]; Proximal northeast of Source Area; shows hydraulic connection to Source Area; closest wells upgradient of CHA property; Total CVOC concentrations are trending upward	VOCs, natural attenuation parameters, total and dissolved metals, qDHC	Bladder*
EA-105S	Well triplet	Water level	n/a
EA-105D	Well triplet	Water level	n/a
EA-105R	Well triplet	Water level	n/a
EA-106D	Six data points[1]; Medial downgradient in north lobe of plume; Compliance monitoring for nearby receptors	VOCs, natural attenuation parameters	Bladder*
EA-106R	Six data points[1]; Medial downgradient in north lobe of plume; Compliance monitoring for nearby receptors	VOCs, natural attenuation parameters	Bladder*
EA-108S	Well triplet	Water level	n/a
EA-108D	Well triplet	Water level	n/a
EA-108R	Well triplet	Water level	n/a
EA-110D	Well couplet	Water level	n/a
EA-110R	Well couplet	Water level	n/a
EA-111D	Well couplet	Water level	n/a
EA-111R	Well couplet	Water level	n/a
EA-117S	Well triplet	Water level	n/a

Well ID (Depth)	Sampling Rationale	Sampling Parameters/	Pump Type
EA-117D	Well triplet	Water level	n/a
EA-117R	Well triplet	Water level	n/a
ME-1	Four data points[2]; Upgradient location; Dubious previous metals results	VOCs, natural attenuation parameters, total and dissolved metals	Bladder
ME-2	Four data points[2]; Proximal downgradient for MNA evaluation; Dubious previous metals results;	VOCs, natural attenuation parameters, total and dissolved metals	Bladder
ME-3	Four data points[2]; Proximal upgradient west of Source Area; Dubious previous metals results	VOCs, natural attenuation parameters, total and dissolved metals	Bladder
ME-4	Four data points[2]; Proximal on south lobe of plume; Dubious previous metals results	VOCs, natural attenuation parameters, total and dissolved metals	Bladder
MW01-06S	Well triplet (with MW01-15D/R)	Water level	n/a
MW01-10S	Well triplet	Water level	n/a
MW01-10D	Well triplet	Water level	n/a
MW01-10R	Well triplet	Water level	n/a
MW01-13D	Two data points[2]; Distal on east lobe of plume; Total CVOC concentrations are trending upward; perimeter well defining plume extent	VOCs, natural attenuation parameters, total and dissolved metals	Bladder
MW01-13R	Two data points[2]; Distal on east lobe of plume; Total CVOCs not detected; perimeter well defining plume extent	VOCs, natural attenuation parameters, total and dissolved metals	Bladder
MW01-13S	One data point; Distal on east lobe of plume; Total CVOCs at trace concentrations; perimeter well defining plume extent	VOCs, natural attenuation parameters, total and dissolved metals	Bladder
MW01-15D	Well triplet (with well MW01-06S)	Water level	n/a
MW01-15R	Well triplet (with well MW01-06S)	Water level	n/a
MW02-03D	Six data points[2]; Distal on east lobe of plume; Total CVOC concentrations are trending upward	VOCs, natural attenuation parameters, total and dissolved metals	Bladder
MW02-03R	Six data points[2]; Distal on east lobe of plume; Total CVOC concentrations are trending upward	VOCs, natural attenuation parameters, total and dissolved metals	Bladder
MW02-03S	One data point; Distal on east lobe of plume; Total CVOCs not detected	VOCs, natural attenuation parameters, total and dissolved metals	Bladder
MW02-08S	Well couplet	Water level	n/a
MW02-08D	Well couplet	Water level	n/a
MW02-11S	Well couplet	Water level	n/a
MW02-11D	Eight data points[2]; Medial between north and east lobes of plume; Total CVOC concentrations are trending upward	VOCs, natural attenuation parameters	Bladder
MW03-02S	Well couplet	Water level	n/a
MW03-02D	Well couplet	Water level	n/a

Well ID (Depth)	Sampling Rationale	Sampling Parameters/	Pump Type
MW03-03D	Nine data points[2]; Medial on east lobe of plume; Total CVOC concentrations are trending downward	VOCs, natural attenuation parameters, total and dissolved metals	Bladder
MW03-03R	Six data points[2]; Medial on east lobe of plume; Total CVOC concentrations are trending downward	VOCs, natural attenuation parameters, total and dissolved metals	Bladder
MW03-03S	Three data points[2]; Medial on east lobe of plume; Total CVOCs not detected	VOCs, natural attenuation parameters, total and dissolved metals	Bladder
MW03-07D	Center of plume	Water level	n/a
MW03-11R	Three data points[2]; Medial on south lobe of plume; Total CVOC concentrations are trending upward	VOCs, natural attenuation parameters, qDHC	Bladder
MW03-11S	Three data points[2]; Medial on south lobe of plume; Total CVOC concentrations are trending upward	VOCs, natural attenuation parameters, qDHC	Bladder
MW03-12D	Seven data points[1]; Medial on east lobe of plume for MNA evaluation; Total CVOC concentrations are trending upward	VOCs, natural attenuation parameters, qDHC	Bladder*
MW03-12R	Six data points[1]; Medial on east lobe of plume for MNA evaluation; Total CVOC concentrations are trending upward	VOCs, natural attenuation parameters	Bladder
MW03-13D	Well couplet	Water level	n/a
MW03-13R	Well couplet	Water level	n/a
MW16-82D	Well couplet	Water level	n/a
MW16-82R	Well couplet	Water level	n/a
MW-DPT-05	Well couplet (with MW-DPT-09)	Water level	n/a
MW-DPT-09	Well couplet (with MW-DPT-05)	Water level	n/a
MW Z2-04	Upgradient of Hall Creek	Water level	n/a
MW Z3-01	Well couplet	Water level	n/a
MW-Z3-01D	Well couplet	Water level	n/a
MW-Z3-02	Northeast corner of plume	Water level	n/a
MW-Z3-03D	Well couplet (with PGU-Z3-06S)	Water level	n/a
NMW-1	Four data points[3]; Proximal west of Source Area; Dubious previous metals results	VOCs, natural attenuation parameters, total and dissolved metals	Bladder
NMW-2	Five data points[2]; Proximal northwest of Source Area; Dubious previous metals results	VOCs, natural attenuation parameters, total and dissolved metals	Bladder
NMW-3	Five data points[2]; Proximal west of Source Area; Dubious previous metals results	VOCs, natural attenuation parameters, total and dissolved metals	Bladder
NMW-4	Five data points[2]; Proximal southwest of Source Area; Dubious previous metals results	VOCs, natural attenuation parameters, total and dissolved metals	Bladder
OB-AS	Single, recent result[1]; Distal on north lobe of plume; perimeter well defining plume extent	VOCs, natural attenuation parameters	Bladder

Well ID (Depth)	Sampling Rationale	Sampling Parameters/	Pump Type
OB-AD	Single, recent result[1]; Distal on north lobe of plume; perimeter well defining plume extent	VOCs, natural attenuation parameters	Bladder
OB-CS	Single, recent result[1]; Distal on south lobe of plume; perimeter well defining plume extent	VOCs, natural attenuation parameters, qDHC , total and dissolved metals	Bladder
OB-CD	Single, recent result[1]; Distal on south lobe of plume; perimeter well defining plume extent	VOCs, natural attenuation parameters, qDHC , total and dissolved metals	Bladder
OB-DS	Single, recent result[1]; Distal on south lobe of plume; perimeter well defining plume extent	VOCs, natural attenuation parameters	Bladder
OB-DD	Single, recent result[1]; Distal on south lobe of plume; perimeter well defining plume extent	VOCs, natural attenuation parameters	Bladder
OB-ED	Two, recent results[2]; Couple with new DPW-MW-3; Medial on south lobe of plume	VOCs, natural attenuation parameters, qDHC	Bladder
OB-FS	Single, recent result[1]; Medial on south lobe of plume; perimeter well defining plume extent	VOCs, natural attenuation parameters, total and dissolved metals	Bladder
OB-FD	Single, recent result[1]; Medial on south lobe of plume; perimeter well defining plume extent	VOCs, natural attenuation parameters, total and dissolved metals	Bladder
OB-GS	Single, recent result[1]; Proximal southwest of Source Area	VOCs, natural attenuation parameters	Bladder
OB-HS	Single, recent result[1]; Distal on south lobe of plume; perimeter well defining plume extent	VOCs, natural attenuation parameters, total and dissolved metals	Bladder
OB-HD	Single, recent result[1]; Distal on south lobe of plume; perimeter well defining plume extent	VOCs, natural attenuation parameters, total and dissolved metals	Bladder
OB-IS	Single, recent result[1]; Distal on south lobe of plume; perimeter well defining plume extent	VOCs, natural attenuation parameters	Bladder
OB-ID	Single, recent result[1]; Distal on south lobe of plume; perimeter well defining plume extent	VOCs, natural attenuation parameters	Bladder
PGU-Z3-02S	Near surface water	Water level	n/a
PGU-Z3-02D	Near surface water	Water level	n/a
PGU-Z3-03S	Near surface water	Water level	n/a
PGU-Z3-03D	Near surface water	Water level	n/a
PGU-Z3-06S	Well couplet (with MW-Z3-03D)	Water level	n/a
PGU-Z3-07S	Well couplet	Water level	n/a
PGU-Z3-07D	Well couplet	Water level	n/a
PGU-Z3-11S	Southeast plume	Water level	n/a
QDC-A-6	South plume	Water level	n/a
QDC-M4	South plume	Water level	n/a
RMW-01S	Well triplet	Water level	n/a
RMW-01D	Well triplet	Water level	n/a

Well ID (Depth)	Sampling Rationale	Sampling Parameters/	Pump Type
RMW-01R	Well triplet	Water level	n/a
SES-01D	Four data points[2]; Total CVOC concentrations are trending upward; Proximal to Source Area	VOCs, natural attenuation parameters, qDHC	Bladder
SES-01R	Four data points[2]; Total CVOC concentrations are trending upward; Proximal to Source Area	VOCs, natural attenuation parameters	Bladder
SES-01S	Four data points[2]; Total CVOC concentrations peaked and fell; Proximal to Source Area	VOCs, natural attenuation parameters	Bladder
SES-09D	Four data points[1]; Total CVOC concentrations are trending upward; Proximal to Source Area	VOCs, natural attenuation parameters	Bladder
SES-09R	Four data points[1]; Total CVOC concentrations are trending upward; Proximal to Source Area	VOCs, natural attenuation parameters	Bladder
SES-09S	Four data points[1]; Total CVOC concentrations are trending downward; Proximal to Source Area	VOCs, natural attenuation parameters	Bladder

1 – The data were predominantly collected during the fall.

2 - Data points are split evenly between spring and fall.

3 – The data were predominantly collected during the spring.

* * - Indicates a dedicated pump is present in the well.

n/a – Pump Type not applicable

2.7.3.3. North Kingstown DPW Shallow Groundwater Investigation

Groundwater samples will be collected from two shallow overburden groundwater monitoring wells to be installed at the North Kingstown DPW Property, in the vicinity of the North Kingstown DPW Building, as depicted on Figure 4. The groundwater samples will be analyzed for VOCs via EPA Method 8260B by the fixed laboratory. The groundwater sampling program for these wells will be executed in a single day. Information regarding the laboratory requirements for groundwater VOCs samples regarding detection limits, precision and accuracy are summarized in Table 5, found in Subsection 2.10, and discussed in Subsection 2.10 and the QAPP (Appendix A).

2.7.4. Sampling Equipment and Procedures

2.7.4.1. Sampling Equipment

- Adjustable rate, bladder pump (constructed of stainless steel with a Teflon bladder)
- Fluorinated ethyl propylene (FEP) tubing, sized appropriate to pump, ¼- or ⅜-inch inner diameter
- Teflon bailer and line (to be used only in the event that low-flow sampling is impossible)
- Solinst water level indicator or oil/water interface probe, as appropriate
- 1-liter graduated cylinder
- Power source for pump, as appropriate
- Water quality meter for pH, SHE-corrected ORP, DO, specific conductance, and temperature,(YSI 699xl) equipped with a flow-through cell

- LaMotte Model 2020e Turbidimeter, or equivalent
- 5-gallon pail
- Low-Flow Groundwater Sampling Log form
- Sample containers (see QAPP Worksheet #19 for sample container requirements).

2.7.4.2. Sampling Procedure - Groundwater

1. Groundwater samples will be collected according to the following procedure, per Stone SOP SEI-6-34.0.
2. Calibrate water quality instruments in accordance with Stone SOPs SEI-5.26.0, SEI-5.28.0, and SEI-5.30.0. Equipment will be calibrated prior to use each day and the calibration will be checked at the end of use on that day to document calibration shifts during use. SHE-correction will be implemented during calibration of the ORP sensor, by calibrating the instrument using the YSI reference electrode value (i.e., 428 mV) at the calibration prompt instead of the Zobell solution value of (i.e., 228 mV).
3. Check for security damage or evidence of tampering on each well, record pertinent observations.
4. Remove well cap and immediately measure VOCs at the rim of the well with a PID instrument and record the reading in the logbook.
5. If the well casing does not have a reference point (usually a V-cut or indelible mark in the well casing), make one. Describe its location and record the date of the mark in the logbook.
6. Check newly constructed wells for the presence of NAPL before the initial sampling round using an oil/water interface probe, in accordance with SOP SEI-6.2.6.
7. Lower pump and any safety cables, tubing and/or electrical lines slowly (to minimize disturbance) into the well to the midpoint of the zone to be sampled. In most instances, the pump will be positioned in the middle of the well screen. For cases where the well screen straddles the water table, the pump should be position 2 feet above the bottom of the well.
8. Before starting pump, measure water level. If a recording pressure transducer is used, initialize starting condition. After recording the height of the water column in the well, determine the volume in gallons by the following formula:

   $$\text{Volume (gallons)} = (\text{Height of water column (ft)} \times \pi \times (\text{Radius (ft)})^2) \times 7.4805 \text{ gallons/ft}^3$$

9. Start pump at its lowest speed setting and slowly increase the speed until discharge occurs. Check water level. Adjust pump speed until there is little or no water drawdown (less than 0.3 feet). If the minimal draw-down that can be achieved is greater than 0.3 feet, continue purging until indicator field parameters stabilize.
10. Discharge purge water into 5-gallon buckets, pending IDW determination (see Subsection 2.15)
11. Monitor and record water level and pumping rate every 3 to 5 minutes (or as appropriate) during purging. Record any pumping rate adjustments (both time and flow rate). Pumping rates should, as needed, be reduced to the minimum capabilities of the pump to ensure stabilization of indicator parameters. Adjustments are best made in the first fifteen minutes of pumping in order

to help minimize purging time. During pump start-up, drawdown may exceed the 0.3 feet target and then "recover' as pump flow adjustments are made.

12. Using the appropriate volumetric container (one that is scalable relative to the volume to be purged), measure the volume of water purged at the same interval as field parameters are recorded (Section 4.3.4). If water is believed to be contaminated, purge water must be containerized. Purge volume calculations should utilize stabilized drawdown value, not the initial drawdown. Do not allow the water level to fall to the intake level (if static water level is above the well screen, avoid lowering the water level into the screen). The final purge volume should be greater than the stabilized drawdown volume plus the extraction tubing volume. This may not be possible in wells with low recharge rates.
13. Wells will be sampled using a bladder pump capable of attaining very low pumping rates, to accommodate wells with low recharge rates. If the recharge rate of the well is lower than the pump's minimum extraction rate, then continue to purge until the drawdown is near (but not below) the top of the well screen. Once the drawdown is within six (6) inches of the top of the well screen, discontinue purging. The well should be sampled as soon as the water level has recovered sufficiently (i.e., 75%) to collect the appropriate volume needed for all anticipated samples (the intake should not be moved during this recovery period). In this case only, samples may be collected even if the well was not purged for four (4) hours and the indicated field parameters did not stabilize.
14. Monitor indicator field parameters (pH, temperature, specific conductance, SHE-corrected ORP, and DO) during purging using a multi-parameter water quality meter equipped with a flow-through cell system. Turbidity will also be monitored, by using an aliquot of groundwater in a separate LaMotte turbidimeter collected upstream of the flow cell. The parameters will be monitored for stabilization prior to collection of the sample. Stabilization is achieved when three consecutive readings, taken at 3- to 5-minute intervals, are within the following limits:
    - Turbidity ± 5 NTU for readings >5 NTU or values <5 NTU
    - DO (± 10% for values greater than 2 milligrams per liter (mg/L); 0.5 mg/L for values less than 2 mg/L)
    - Specific conductance ± 3%
    - Temperature ± 3%
    - pH (± 0.1)
    - SHE-corrected ORP (± 10 mV)
    - Drawdown (no more than 0.3 feet)
15. The low-flow guidance suggests a maximum drawdown of 0.3 feet. However, in fractured bedrock, it is not uncommon to observe greater drawdown under low flow pumping conditions. The hydraulic head in the interval will be carefully monitored and a purge rate used that will minimize drawdown. If head continues to decline, the rate of decline will be calculated. Once this rate has stabilized for three consecutive readings, this criterion will have been satisfied.

16. Upon stabilization, collect water samples for laboratory analyses from a sample port located in front of the flow-through-cell (use a by-pass assembly or disconnect cell to obtain the sample).
17. VOC samples should be collected first and directly into the sample containers, using a zero headspace vial holder located on the suction side of the pump. Sample water should be allowed to fill sample containers gently down the inside of the container with minimal turbulence. For samples collected for VOC analysis, use pre-preserved 40-mL glass vials with Teflon septa, ensuring no headspace in the closed sample container.
18. During purging and sampling, the tubing should remain filled with water so as to minimize possible changes in water chemistry upon contact with the atmosphere. If the pump tubing is not completely filled to the sampling point, use one of the following procedures to collect samples:
    a. Add a clamp, connector (Teflon or stainless steel), or valve to constrict the sampling end of tubing; or
    b. Insert small diameter Teflon tubing into water filled portion of pump tubing, allowing the end to protrude beyond the end of the pump tubing, collect samples from small diameter tubing.
19. Secure the well.
20. At a minimum, well purging times and/or volumes, water level in the well before and after pumping, calculation of one well volume, weather conditions (temperature, cloud cover, wind direction and recent precipitation events) and sample collection time will be recorded for each sampled well on the field forms provided with Stone SOP SEI-6-34.0.

2.7.4.3. Sampling Procedure – Non-Aqueous Phase Liquid

1. NAPL samples will be collected according to the following procedure, per Stone SOP SEI-6-47.0.
2. Check for security damage or evidence of tampering on each well, record pertinent observations.
3. Remove well cap and immediately measure VOCs at the rim of the well with a PID or FID instrument and record the reading in the logbook.
4. If the well casing does not have a reference point (usually a V-cut or indelible mark in the well casing), make one. Describe its location and record the date of the mark in the logbook.
5. Confirm the presence and thickness of NAPL before the initial sampling round using an oil/water interface probe, in accordance with SOP SEI-6.2.6.
6. Depending on the thickness of NAPL present select one of the following sampling tools:
    a. For a foot or more of NAPL, use a peristaltic pump with Teflon tubing; or
    b. Use a Teflon bailer and line.
7. Insert the sampler (either tubing or bailer) to the NAPL interval of the well. Activate the pump or withdraw the bailer to retrieve the NAPL.

8. Discharge the NAPL into appropriate sample containers. For samples collected for VOC analysis, use pre-preserved 40-mL glass vials with Teflon septa, minimizing headspace in the closed sample container.
9. Upon completion of sampling, secure the well.

2.8. Residential Well Water Sampling

2.8.1. Site Background

The Rhode Island Department of Health (RIDOH) sampled eight private water supply wells for VOCs in 1997. The five wells located closest to the PR-58 Property had no detections of VOCs, nor did the two northeasternmost private wells along Fletcher Road. Trace concentrations of PCE (0.7 µg/L) and 1,1,1-TCA (1 µg/L) (1,1,1-TCA is not a PR-58-related contaminant) were measured in the sample collected from a well (WS-4) located more than 2,000 feet north-northeast of the Property at 550 Fletcher Road. In 2006, USACE requested permission to resample the eight wells and was granted permission to sample only three. The three private water supply wells (WS-6, WS-9, and WS-18) for which access for sampling was granted were sampled in September 2006. The wells were located at residences along Fletcher Road. No CVOCs were detected in the samples from WS-6 and WS-18. The only VOC detected in the sample from WS-9 was chloromethane (not a PR-58-related contaminant). In 2009, access was granted to sample seven private water supply wells: three wells located on Fletcher Road that had been sampled previously in 1997 and 2006 (WS-6, WS-9, and WS-18), and four new wells, including three located on Potter Road. However, permission has not granted to sample WS-4 since 2006, so conditions at this and other locations where access has been denied have not been updated.

2.8.2. Sampling Objectives

The objective of this task is to obtain representative well water samples from select private water wells. It is the responsibility of USACE to obtain access to the residential drinking water wells for sampling and the responsibility of Stone to research which properties may have wells, contact and communicate with property owners to determine which ROE's USACE needs to obtain.

The data obtained from these tasks should be of sufficient quality to allow the project team to make decisions regarding potential impact to drinking water receptors. The screening levels used for this project are RIDEM groundwater standards and USEPA MCLs.

2.8.3. Sample Location and Frequency

Table 4 lists the two private wells, neither used for drinking purposes, which are proposed for well water sampling as part of the RI/FS investigation:

Table 4: Residential Water Wells Proposed for Sampling

Residence Address	Well Identification
710 Fletcher Road	WS-25
772 Fletcher Road	WS-26

The approximate locations of the wells proposed for well water sampling as part of the RI/FS investigation are depicted on Figure 6. The locations of the wells will be recorded using a survey-grade GPS during the sampling event. The well water samples will be analyzed for CVOCs via EPA Method 524.2 by the fixed laboratory. The well water sampling program will be executed in a single day. Information regarding the laboratory requirements for well water CVOCs samples regarding detection limits, precision and accuracy are summarized in Table 5, found in Subsection 2.10, and discussed in Subsection 2.10 and the QAPP (Appendix A).

2.8.4. Sampling Equipment and Procedures

2.8.4.1. Sampling Equipment

- Sample labels
- Indelible pen/marker
- Thermometer
- Field logbook
- Sample containers (see QAPP Worksheet #19 for sample container requirements).

2.8.4.2. Sampling Procedures

1. Well water samples will be collected according to the following procedure, per Stone SOP SEI-6-44.0.
2. Obtain access to the specified sampling location. Be sure to bring a copy of the Access Letter.
3. Open the sampling port. For interior ports this may require the use of a hose or bucket to remove purge water. Remove the aerator from the tap.
4. Allow the water to run freely until distribution system is flushed. This is most conveniently determined by monitoring temperature of the water, as well as listening for the activation of the water pump to replenish the supply's pressure tank. Flush the system for 10 minutes or until the temperature of the water stabilizes, whichever is later. Values are to be recorded on appropriate field forms or on Observation and Remark forms. Purging is considered complete and sampling may begin when all the above indicator field parameters have stabilized. Stabilization is considered to be achieved when three (3) consecutive temperature readings, taken at three (3) to five (5) minute intervals, are within 3%.
5. For samples collected for VOC analysis, use pre-preserved 40-mL glass vials with Teflon septa, ensuring no headspace in the closed sample container. Place samples in a cooler chilled to <6ºC until delivered to the analytical laboratory.
6. Document the collection of all samples in the field notebook in accordance with SEI SOP 4.5.n, most current version and the QAPP. At a minimum, include the following information: location, date, time, exact sample station, time flushed, stabilization temperature, the number of samples collected, and any unusual observations or events.

2.9. Sample Identification

2.9.1. Site Background

As part of RI/FS Task Numbers 1 through 7, samples will be collected for laboratory analysis. Each of these activities will require identifying prior to sample handling and analysis.

2.9.2. Sampling Objectives

The objective of this procedure is to accurately assign each sample with a unique identifier.

The identifiers assigned as part of these tasks should allow for the accurate tracking of sample location, depth, and matrix.

2.9.3. Sample Location and Frequency

The sample locations for each area investigated as part of the project are depicted in Figures 2 through 6. Each sample collected as part of this investigation will be assigned a unique identifier.

2.9.4. Sampling Procedures

1. Each sample will be assigned a unique sample identifier shown on the COC and container labels.
2. The first set of characters will be the alphanumeric location identification (ID) which will identify the sample location. Samples from existing wells will use the most recent sample name for that well. All other locations will be assigned an individual consecutive number preceded by a letter code for the type of sample:
    - "SA" for Source Area overburden soil sampling;
    - "HC" for pore water, surface water, and sediment investigations associated with Hall Creek;
    - "NW" for pore water, surface water, and sediment investigations associated with the northern wetlands;
    - "SEI" for samples collected from the Town of North Kingstown Department of Public Works property wells SEI-1 and -2 and Source Area wells SEI-3 to SEI-5; and
    - "RES" for residential well water samples.

    The location ID code will be followed by a dash (e.g., "SA1-").
3. The second set of characters will identify the depth interval from which the sample was collected. The actual depth will be used as rounded to the nearest 0.1 foot. The depth interval shall be the depth of the sample in fbgs. For surface water samples the depth will be the depth below the water surface. If the sample interval sequence is not valid or unknown, an "X" will be used. The interval code will also be followed by a dash.

    The last character(s) will be a letter(s) identifying the sample matrix:
    - "GW" Groundwater sample
    - "SW" Surface water

- "PW" Pore water
- "SD" Sediment
- "SO" Soil sample
- "D" Field duplicate
- "EB" Equipment blank
- "TB" Trip Blank
- "MS" Matrix spike sample
- "MSD" Matrix spike duplicate sample
- "IDW" Investigation Derived Waste sample

Examples of sample IDs are as follows:

- Sample SA1-5.0-SO is a soil sample from the first investigation location SA1. The soil was collected beginning from a depth of 5.0 fbgs.
- Equipment blanks will be identified as "EB" in relation to the sample location tested immediately prior to their collection/submittal to the onsite laboratory. For instance OB1-X-EB identifies an equipment blank collected after the testing of location OB1.
- Trip blanks will be identified as "TB" followed by the date that they sent to the lab with the associated samples. For multiple trip blanks sent on the same day, sequential alphabetic letters were added to the identification; e.g. TB-091609A, TB-091609B, etc.

2.10. Sample Handling and Analysis

2.10.1. Site Background

As part of RI/FS Task Numbers 1 through 7, samples will be collected for laboratory analysis. Each of these activities will require sample handling and analysis.

2.10.2. Sampling Objectives

The objective of this procedure is to identify and manage:

1. The types of environmental samples collected;
2. The analytical methods that will be used and a listing of their analyte detection limits against the appropriate enforcement standards; and
3. The types and quantities of QC samples that will be used to evaluate the quality (precision and accuracy) of the data.

2.10.3. Sample Location and Frequency

To meet the DQOs that are stated for each of the various RI/FS tasks in Subsections 2.1 through 2.8, the following sampling and analytical criteria have been selected and are shown on Table 5. Table 5 summarizes the types of media being sampled, the analytical methods and the type and quantity of QC samples that will be

collected as part of this work. Two types of laboratories will be used for this work, an onsite laboratory and a fixed laboratory.

Table 5: Laboratory Analysis Methods and Expected Numbers of QC Samples

Matrix	Sample Source/Type	Parameter	Analytical Method	Type of Laboratory	# Field Samples	QC Samples			Total Samples
						Duplicate	Field/Trip Blanks	MS/MSD	
Water	Existing Monitoring Wells	VOCs	8260B	Fixed	53	3	4/4	3	67
		Total Metals	6010/7470/7000	Fixed	26	2	2/0	2	32
		Dissolved Metals	6010/7470/7000	Fixed	26	2	20	2	32
		Ethane/ethene	RSK 175	Fixed	51	3	4/4	3	65
		TOC	9060A	Fixed	51	3	0	0	54
		qDHC, tceA, bvcA and vcrA[3, 4]	Proprietary	Fixed	10	1	0	0	11
		Nitrate/nitrite[3] sulfate/sulfite[3] ferrous/ferric iron[3] chloride[3]	HACH Field Methods *Spectrophotometric* Nitrate – 8171 Nitrite – 8507 Sulfate – 8051 Sulfite – 8071 Ferrous iron – 8146 Total iron – 8008 *Titration* Chloride - 8225	Field	51	3	0	3	57
	DPW Shallow Monitoring Wells	VOCs	8260B	Fixed	2	1	1/1	1	6
	Pore Water	VOCs	8260B	Fixed	13	1	1/1	1	17
		Total Metals	6010/7470/7000	Fixed	7	1	1/0	1	10
		Dissolved Metals	6010/7470/7000	Fixed	7	1	1/0	1	10

Table 5: Laboratory Analysis Methods and Expected Numbers of QC Samples

Matrix	Sample Source/Type	Parameter	Analytical Method	Type of Laboratory	# Field Samples	QC Samples			Total Samples
						Duplicate	Field/Trip Blanks	MS/MSD	
	Surface Water	VOCs	8260B	Fixed	13	1	1/1 [1]	1 [1]	17
		Hardness	6010	Fixed	13	1	1/0	1	16
		Total Metals	6010/7470/7000	Fixed	7	1	1/0	1	10
		Dissolved Metals	6010/7470/7000	Fixed	7	1	1/0	1	10
	Residential Well Water	VOCs	524.2	Fixed	2	1	1/1	1	6
	IDW	VOCs	8260B	Fixed	TBD [2]	0	0	0	TBD [2]
		SVOCs	8270	Fixed	TBD [2]	0	0	0	TBD [2]
		Pesticides	80810	Fixed	TBD [2]	0	0	0	TBD [2]
		PCBs	8082	Fixed	TBD [2]	0	0	0	TBD [2]
		Metals	6010/7470/7000	Fixed	TBD [2]	0	0	0	TBD [2]
NAPL	NAPL	VOCs	8260B	Fixed	3	0	1	0	4
		SVOCs	8270	Fixed	3	0	0	0	3
		TPH	8100M	Fixed	3	0	0	0	3
		PCBs	8082	Fixed	3	0	0	0	3
		Petroleum Finger Print	8015M	Fixed	3	0	0	0	3

Table 5: Laboratory Analysis Methods and Expected Numbers of QC Samples

Matrix	Sample Source/Type	Parameter	Analytical Method	Type of Laboratory	# Field Samples	QC Samples			Total Samples
						Duplicate	Field/Trip Blanks	MS/MSD	
		Viscosity [3]	ASTM D 445 (@40°C)	Fixed	3	0	0	0	3
		Specific Gravity [3]	ASTM D 1298	Fixed	3	0	0	0	3
Soil	Sediments	VOCs	8260B Modified	Fixed	13	1	1/1 [1]	1	17
		TOC	Lloyd Kahn Method	Fixed	13	1	1/0	0	15
	Soil Cores	VOCs	8260C Modified	Onsite	512	26	10/11	26	585
		Porosity & Density	ASTM D7263	Fixed	10	0	0	0	10
		FOC	ASTM D 2974	Fixed	10	1	0	0	11
		TPH	8100M	Fixed	10	1	1/1	1	14
	IDW	VOCs	TBD [2]	Fixed	TBD [2]	0	0	0	TBD [2]
		SVOCs	8270	Fixed	TBD [2]	0	0	0	TBD [2]
		Pesticides	80810	Fixed	TBD [2]	0	0	0	TBD [2]
		PCBs	8082	Fixed	TBD [2]	0	0	0	TBD [2]
		Metals	6010/7470/7000	Fixed	TBD [2]	0	0	0	TBD [2]
Rock	Bedrock	porosity	API R-40 Guidelines	Fixed	12	0	0	0	12

TBD indicates that the number is to be determined at a later date after consultation with USACE, RIDEM and USEPA.

Note 1: The surface water and sediment samples will be in the same sample delivery group as the pore water samples.

Note 2: IDW is investigation derived waste samples for characterizing waste for offsite transport and disposal. The number of samples is to be determined pending the volume of water and soil

Table 5: Laboratory Analysis Methods and Expected Numbers of QC Samples

Matrix	Sample Source/Type	Parameter	Analytical Method	Type of Laboratory	# Field Samples	QC Samples			Total Samples
						Duplicate	Field/Trip Blanks	MS/MSD	

generated, and type of analyses required.

Note 3: Laboratory is not DOD certified for this method.

Note 4: Proprietary analytical method.

2.10.4. Sampling Equipment and Procedures

Sample handling and transportation procedures will be conducted in accordance with Stone SOP SEI-6.16.4, and are summarized in QAPP Worksheets # 26 and 27.

Samples will be maintained under chain of custody (CoC) in accordance with Stone SOP SEI-4.2.7. The samples will be kept in the possession of the sampler until they can be securely packaged. For samples shipped to the fixed laboratory, the sampler will seal the samples in a sturdy shipping box or other suitable container. The sampler will complete the CoC form, and insert the CoC form into a ziplock bag attached to the lid inside of the shipping container such that the form can be filled out and signed each time the samples change possession, from sampler, to courier, to laboratory, as appropriate. Details are included in are summarized in QAPP Worksheets # 26, 27, and 29.

Data will be handled in accordance with Stone SOP SEI-4.5.11.

2.10.4.1. Onsite Laboratory

The onsite laboratory will be used for two purposes:

1. To provide near-real time VOC analytical data to support the delineation of the vertical and horizontal extent of VOC contamination in the overburden soils; and
2. To measure in a timely manner the natural attenuation parameters during the monitoring wells sampling phase.

The onsite laboratory will analyze soil samples for VOCs using a Solid Phase Microextraction (SPME) sample preparation technique followed by analyses on a Hewlett Packard 5890 Series II, equipped with a Mass Spectrometer (MS). Field Laboratory Analysis will be performed in accordance with Stone SOP SEI-10.15.9 "The Determination of Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) (SW846 US EPA Method 8260C), 7 November 2012. An unrestricted copy of this method is provided in Attachment C. For the onsite laboratory analyses, results will be reported strictly to the Limits of Quantitation (LOQ). The LOQs by SW846 Selective Ion Monitoring (SIM) Method 8260C modified with SPME are 80 µg/Kg for soil. The field laboratory, using Method 8260C, will provide data only for a limited list of primary contaminants of potential concern (COPC): PCE; TCE; tetrachloroethane (TeCA); 1,1,2-TCA; cis-1,2-DCE; trans-1,2-DCE; 1,1-DCE; and VC. In addition, since the chemical composition of the reported NAPL is unknown, the target analyte list will be expanded to include the following petroleum VOCs: benzene; toluene; ethylbenzene; total xylenes; 1,2,4-trimethylbenzene (TMB); 1,3,5-TMB, and naphthalene. PCA, PCE, TCE, 1,1,2-TCA, cis- and trans- 1,2-DCE, 1,1-DCE, 1,1-DCA, 1,2-DCA, and VC (see Table 5).

The onsite laboratory will provide data only for the limited list of CVOCs/COPC listed above which are the Site-related compounds. By limiting the compound list, the field analyses will be quicker, and the relevant field decisions can therefore be made more effectively. However, the onsite laboratory will also report, as tentatively identified compounds (TICs), non-target compounds that are encountered in the groundwater samples. The onsite laboratory uses a SIM mass spectrometer program, as opposed to a full-scan mass spectrometer program; therefore, the spectral information is limited to the ions selected for the SIM method. The selected ions are typically the same as the primary and qualifier ions that are recommended in the EPA's SW846 8260C method. The non-target compounds that can be identified by the onsite lab are listed in the onsite laboratory's SOP for Method 8260C. QAPP Worksheet #15-A summarizes the onsite laboratory VOC target analytes, detection/reporting limits and regulatory limits.

Several of the natural attenuation parameters (i.e. nitrite and ferrous iron) require near-real time analyses as they are short-lived (i.e., ferrous iron converts to ferric iron within minutes of being exposed to oxygen) outside of their natural environment. The natural attenuation parameters will be analyzed as soon as the samples are collected from the monitoring wells using a combination of field-based wet chemistry and spectrophotometric methods. The methodology for these procedures is found in the Hach *Water Chemistry Handbook, 5th Edition*. The pertinent portions of the reference can be found in Attachment B. QAPP Worksheet #15-B summarizes the field analytical detection/reporting limits and regulatory limits.

Data reporting schedules and requirements for the onsite laboratory are covered in Subsection 6.2.1 of the Work Plan.

2.10.4.2. Fixed Laboratory

A fixed laboratory will be used for all other analyses conducted as part of this work, as shown on Table 5. The fixed (offsite) laboratory will be used for the following purposes:

1. To provide VOC analytical results for pore water, surface water, and sediment samples, hardness analytical results for surface water, and TOC analytical results for sediments, to evaluate possible impacts to the Hall Creek system;
2. To provide VOC analytical results for groundwater samples to extend the trend analysis of the existing groundwater analytical data set;
3. To provide total and dissolved metals analytical results for groundwater samples to evaluate the representativeness of previous analytical results;
4. To provide general groundwater chemistry analytical data (ethane/ethenes, and TOC) to support the evaluation of natural attenuation;
5. To provide microbiological data (tceA, bvcA and vcrA3, plus qDHC) to support the evaluation of natural attenuation;
6. To provide additional analytical results (porosity, density, FOC, and TPH) to supplement the VOC analytical results for Source Area investigation soil samples provided by the onsite laboratory;
7. To provide NAPL analysis (VOCs, SVOCs, TPH, PCBs, fuel fingerprint, viscosity, and specific gravity);
8. To provide VOC analytical results for groundwater samples to evaluate the soil vapor migration pathway at the North Kingstown DPW Property;
9. To provide low-concentration analytical results for drinking water samples collected from nearby residential properties; and
10. To determine bedrock porosity.

ESS Laboratory of Cranston, RI will be the primary fixed lab used for these analyses. ESS is DOD-accredited for the analyses required for this project, with a few exceptions as noted on Table 5. The fixed laboratory SOPs are contained in Attachment C. Quality control samples such as duplicates, matrix spikes, field blanks and trip blanks will be prepared and analyzed. ESS will subcontract several of the analyses to TestAmerica and Microbial Insights Laboratories and others. ESS Laboratory uses a modification to EPA Methods 8100M and

8015M, which changes to the oils used to calibrate the instrument, from oils found at the site to aliphatic hydrocarbons from the lab. For EPA Method 8100M, ESS Laboratory uses a GC/Flame Ionization Detector to evaluate total petroleum hydrocarbons in the C_9-C_{36} range. Data reporting schedules and requirements for the fixed laboratory are covered in Section 6.2.2 of the Work Plan.

Worksheets #15-C and 15-D of the QAPP summarize the fixed laboratory VOC target analytes and TAL Metals analytes, limits of detection (LODs)/ LOQs and regulatory limits for groundwater samples. Worksheets #15-E through 15-I of the QAPP summarize the fixed laboratory LODs/LOQs for the remaining groundwater sample analyses, for which there are no regulatory limits.

Worksheet #15-J of the QAPP summarizes the fixed laboratory LODs/LOQs for hardness in surface water sample analyses, for which there are no regulatory limits. Worksheets #15-K and 15-L of the QAPP summarize the fixed laboratory VOC target analytes, LODs/LOQs and regulatory limits for pore water, surface water, and sediment samples. Worksheet #15-M of the QAPP summarizes the fixed laboratory LODs/LOQs for TOC for sediment sample analyses, for which there are no regulatory limits.

Worksheets #15-N through 15-P of the QAPP summarize the fixed laboratory porosity, density, FOC, and TPH analytes, LODs/LOQs and regulatory limits, for which there are no regulatory limits, for soil samples. Worksheet #15-Q of the QAPP summarizes the fixed laboratory LODs/LOQs for porosity in rock sample analysis, for which there are no regulatory limits.

Worksheets #15-R through 15-Y of the QAPP summarize the fixed laboratory VOC, SVOC, Pesticide, PCB, TAL Metals, TPH & petroleum fingerprint, viscosity and specific gravity analyses LODs/LOQs and regulatory limits for which there are no regulatory limits, for NAPL and IDW samples, respectively.

2.11. Utility Clearance

2.11.1. Site Background

As part of RI/FS Field Task Number 4, about 30 soil borings will be advanced, and three NAPL monitoring wells may be installed, within the Source Area. As part of Field Task Number 5, two shallow overburden monitoring wells will be installed in soil borings advanced on the North Kingstown DPW Property. A component of both of these activities will require clearing potential soil boring locations for underground utilities.

2.11.2. Sampling Objectives

The objective of this procedure is to assure that soil boring activities are conducted in areas free of underground utilities.

The successful achievement of this objective will result in no damage to subsurface utilities or injury to drilling equipment or personnel during subsurface investigations at the Site. The data analyses from this sampling method/procedure will be generated as soon as possible after data collection so that the information can be used with the re-processed geophysical data, aerial photo interpretation, bedrock outcrop data and groundwater profiling data to select the locations for new monitoring wells.

2.11.3. Sample Location and Frequency

The following areas will be subject to utility clearance as part of the RI/FS investigation:

- The location of all soil borings and up to three NAPL monitoring wells in the vicinity of well EA-102D, depicted in Figure 3; and,
- The location of soil borings for two shallow overburden groundwater monitoring wells to be installed at the North Kingstown DPW Property, depicted in Figure 4.
- To the extent possible, Stone will coordinate subsurface utility clearance with other contractors working at nearby Navy sites, in order to maximize resources.

2.11.4. Sampling Equipment and Procedures

Geophysical investigations will be performed by Stone subcontractor Hager-Richter Geoscience, Inc., using *Electromagnetic Induction, Ground Penetrating Radar and Precision Utility Location.* Detail regarding surface geophysical techniques and procedures are provided in the text below.

2.11.4.1. Sampling Equipment

- Geonics EM61-MK2 time domain electromagnetic induction metal detector
- Sensors and Software Noggin Plus digital ground-penetrating radar (GPR) system, using a survey wheel to trigger recording of the data at equal intervals and a 250 megaHertz (MHz) antenna
- Radiodetection RD4000 series precision electromagnetic pipe and cable locator
- CST FT-70 magnetic pipe locator

2.11.4.2. Sampling Procedure

1. Available as-built drawings and subsurface utility information from the property owners (e.g., QDC and North Kingstown DPW) will be reviewed by Stone and provided to the geophysics subcontractor prior to the location survey. In addition, DigSafe utility clearance will be performed in advance of the subsurface utility clearance, in order to provide guidance to the geophysics subcontractor.
2. The survey areas will be cleared of moveable obstructions and brush prior to the survey.
3. A local 5-ft grid will be established, centered on the locations of the proposed soil borings, and the entire area within the Source Area investigation will be cleared for utilities, to provide for maximum sample location flexibility.
4. The corners of the survey grid will be surveyed using GPS and noted on the Clearance Summary Sheets.
5. EM61 data and GPR data will be acquired along survey lines spaced no more than 5-feet apart in the accessible portions of the survey area.
6. The PUL equipment will used in passive mode to sweep the area of interest for utilities carrying live electrical current. If possible, connections are made to nearby utilities to determine if they enter the area of interest.

7. The data will be processed in the field and the interpretation will be marked on the ground at the Site and marked on the Clearance Summary Sheets for transmittal to the client at the end of each work day.

2.12. Site Survey

2.12.1. Site Background

As part of RI/FS Task Numbers 2 and 3, a staff gauge will be installed in the northern wetland, and staff gauges along Hall Creek may need to be reset and surveyed. As part of Field Task Number 4, up to 3 new NAPL monitoring wells may be installed in the Source Area. As part of Task Number 5, three shallow overburden monitoring wells will be installed on the North Kingstown DPW Property. A component of all three of these activities will require obtaining water level measurements.

2.12.2. Sampling Objectives

The objective of this procedure is to accurately measure the depth to water in overburden monitoring wells or NAPL monitoring wells.

The measurements obtained as part of these tasks should meet industry standards, namely, ASTM D4750, and be accurate to 0.01 ft, as well as be certified by a Rhode Island-licensed surveyor.

2.12.3. Sample Location and Frequency

The following new wells will be subject to location and elevation surveying as part of the RI/FS investigation:

- Up to three NAPL monitoring wells in the vicinity of well EA-102D, depicted on Figure 3; and
- Up to two newly-installed shallow overburden groundwater monitoring wells at the North Kingstown DPW Property, depicted on Figure 4.

2.12.4. Sampling Equipment and Procedures

2.12.4.1. Sampling Equipment

Surveying of the locations of the soil borings and wells associated with the Source Area investigation will be performed using traditional transit-and-rod methodology, due to the high density of the locations. The other locations requiring surveying will be surveyed using digital global positioning methods, due to their separation from each other and available benchmarks. The following equipment will be used.

1. Transit
2. Tripod
3. Stadia rod
4. Survey-grade GPS
5. Field forms or other appropriate media.

2.12.4.2. Sampling Procedure

The following procedure describes the method to determine relative field elevations for the Source Area investigation, per Stone SOP SEI-6.23.1. The digital surveying methods will be performed according to the manufacturer's specifications. All map coordinates for this project are referenced to the horizontal datum: Rhode Island State Plane Coordinate System Northing and Easting (coordinates in feet), North American Datum of 1983 (NAD83); and the North American Vertical Datum of 1988 (NAVD88).

The northing and easting of each sample location (borings, wells, etc.) will be determined to an accuracy of ± 0.1 foot. The ground surface elevation, and top of inner and outer casing will be measured to the nearest 0.01 foot and reported to the nearest 0.1 foot referenced to the NAVD88 datum (in feet) using existing on-Site benchmark elevations provided by USACE. Stone's surveyor will be responsible for establishing appropriate survey control. Top-of-casing elevations will be measured to the nearest 0.01 foot. All surveying measurements will be performed by a Rhode Island licensed surveyor after all sampling locations are determined and will meet the USACE positional data quality requirements (EM 1110-1-1005, 2007 version (USACE, 2007)).

1. Set up a table in the field notebook or on field data sheets with columns labeled as point location, backshot (+), foreshot (-), and elevation.
2. Set the tripod up so as to have direct line-of-sight to as many points to be measured as possible, in order to minimize set-ups.
3. Level the instrument according to the manufacturer's instructions.
4. The stadia rod is to be held vertically over the measuring point of a piezometer (monitoring well) or on a point of some known elevation. Note that this measuring point is the same point from which water levels in the piezometers (monitoring wells) are to be measured. If there is no mark, one should be made in black indelible ink. Be very careful to keep dirt or other debris from falling into the well. It may be necessary to cover the well with a plastic baggie for protection.
5. With the instrument leveled, read the measurement on the stadia at the middle cross hair. Record this measurement as the backshot (+).
6. Move the stadia rod to the next position beyond the instrument roughly the same distance as the backshot. This is designated as a turning point (could be the next piezometer or monitoring well).
7. Pivot the level around being careful not to disturb the tripod. Read the stadia rod and record as the foreshot (-).
8. Move the instrument with its tripod and level, roughly the same distance beyond the stadia rod as before (this can be done by pacing).
9. Pivot the stadia rod being careful not to change the elevation.
10. Read the measurement on the stadia rod at the turning point. Record as the backshot.
11. Repeat steps 6 through 10 working around the field to close the loop until the last foreshot is made at the original starting point.

2.13. Water Level Measurement

2.13.1. Site Background

As part of RI/FS Task Number 3, groundwater samples will be collected from approximately 51 monitoring wells at the Site. As part of RI/FS Task Number 4, NAPL monitoring wells may be installed in the Source Area. As part of Task Number 5, two shallow overburden monitoring wells will be installed on the North Kingstown DPW Property. Finally, as part of Task Number 2, surface water samples will be collected from the Hall Creek drainage and a wetland located north of the Property, for which surface water elevations will be recorded. As part of Task Number 30, groundwater elevation measurements will be recorded from approximately 50 monitoring wells at the Site, in addition to the wells described previously in this subsection. A component of all of these activities will require obtaining water level measurements.

2.13.2. Sampling Objectives

The objective of this procedure is to accurately measure the depth to water in overburden monitoring wells or NAPL monitoring wells.

The measurements obtained as part of these tasks should meet industry standards, namely, ASTM D4750, and be accurate to 0.01 ft.

2.13.3. Sample Location and Frequency

The following wells will be subject to water level measurement as part of the RI/FS investigation:

- Up to three NAPL monitoring wells may be sampled in the vicinity of well EA-102D, depicted on Figure 3;
- Up to 51 groundwater monitoring wells across the Site, depicted on Figures 5a and 5b, and summarized in Table 3;
- Up to two shallow overburden groundwater monitoring wells will be sampled at the North Kingstown DPW Property, depicted on Figure 4;
- Two existing staff gauges located within the Hall Creek system, and one staff gauge to be set for the northern wetland location; and
- .Up to 50 additional groundwater monitoring wells across the Site, depicted on Figures 5a and 5b, and summarized in Table 3.

2.13.4. Sampling Equipment and Procedures

- Map of location(s) of well(s) to be measured.
- Keys to locked wells.
- Proper protective clothing (if necessary or as designated in the protocol or QAPP).
- Field notebook or other acceptable recording medium.
- Water level measuring device (electronic water indicator, interface probe, or other device).
- Sampling gloves.

- Decontamination materials, as necessary.
- Clean (new) polyethylene/plastic sheeting or other material on which well cap and other equipment may be placed.

2.13.5. Sampling Procedure

1. The procedures listed below are consistent with Stone SOP SEI-6.2.6.
2. Locate wells on the Site map.
3. Unlock and remove the well guard if one is present, then remove the well cap. Unless the inner casing is vented, this should be done well in advance of the measurement of the depth to water in order to allow the water level to equilibrate once the cap is off. Be careful to prevent the inside of the well cap from touching the ground in order to prevent possible contamination. Clean, poly/plastic sheeting (or other appropriate material) may be spread on the ground around the well to set the cap on.
4. Determine the established measuring point, which should be marked on the well casing with indelible ink. If no mark exists, establish a measuring point at the highest point on the rim of the well casing and mark it with indelible ink. The measuring point must be on the well casing itself and not on a separate well guard, since the well guard may shift due to ground surface settling or frost heaves. If a pump head assembly is dedicated to the well, a mark should be established on the head assembly (near the access port).
5. Prior to inserting anything down the well, be sure to don clean sampling gloves and to decontaminate the measuring device.
6. Whenever possible, use an electric water level indicator to measure the water level. The water indicator consists of a graduated electric cable with a weighted probe at the end. The probe consists of an open electric connection; when the probe come in contact with the water, the connection is closed and a current travels through the wire, causing a signal to be activated. The signal may be an audible alarm or a light that is turned on. Lower the probe into the well slowly until the signal is noted. Put the graduated wire against the measurement point, then raise and lower the probe with decreasing increments to determine precisely where the water surface is.
7. Record in the field notebook or appropriate form the water level based on the graduation of the tape; typically this is to the nearest 0.01 foot. If a NAPL is known or expected to be present, the measurements shall be made with an interface probe. The probe is lowered slowly into the well until a liquid is encountered. The probe signifies a low conductance liquid such as NAPL with a continuous tone. The probe is then slowly lowered deeper into the well to determine the position of the NAPL/water interface. The presence of a high conductance fluid such as water is indicated by an intermittent tone.
8. Other measuring devices, such as a measuring tape or a string, may be used to make approximate water level measurements, but they should be used only if an electronic water indicator is not available.
9. Note the water level, and the NAPL level if applicable, in a field notebook or other acceptable medium as a measurement below the top of the well casing (BTOC). Also note the time of the water level measurement.

10. Decontaminate the measuring instrument after each measurement in accordance with SOP SEI-5.l.n.
11. The sequence of water level measurements shall be from least likely contaminated to most likely contaminated sampling points, or if contamination levels are unknown, from upgradient to downgradient locations, in order to reduce the potential for cross-contamination.
12. Once the water level is measured, and the measurement recorded, the well cap is then set back on the well casing, and the locking well guard re-locked (if applicable). If the lock or guard needs lubrication, be sure to take appropriate steps to prevent any possible contamination of the site, well, or well casing.

2.14. Maintenance and Decontamination of Field Equipment

2.14.1. Site Background

As part of each RI/FS Task, field equipment will be used, and will come in contact with potentially-contaminated media. Therefore, field equipment will require maintenance and decontamination.

2.14.2. Sampling Objectives

The objective of this procedure is to maintain and decontaminate field equipment, so that it can be relied upon to collect high-quality data.

Proper maintenance and decontamination of field equipment is an essential quality control measure and a safety precaution to ensure sampling equipment is in proper working condition and to remove any chemical or material contamination. The procedure prevents cross-contamination between sampling stations and helps maintain a clean working environment for the safety of all field personnel. Equipment decontamination helps prevent sampling bias.

2.14.3. Sample Location and Frequency

Field equipment, such as water quality meters and water level indicators, used in all parts of the RI/FS investigation will require maintenance and decontamination. Equipment maintenance will be performed as necessary; equipment decontamination will be performed before equipment use, between sampling locations/intervals, and at the conclusion of the RI/FS investigation. A separate decontamination area will be established at the North Kingstown DPW property, to eliminate the need to transport potentially-contaminated equipment back to the main area prior to decontamination.

2.14.4. Sampling Equipment and Procedures

- Map of location(s) of well(s) to be measured.
- Keys to locked wells.
- Proper protective clothing (if necessary or as designated in the protocol or QAPP).
- Field notebook or other acceptable recording medium.
- Water level measuring device (electronic water indicator, interface probe, or other device).
- Sampling gloves.

- Decontamination materials, as necessary.
- Clean (new) polyethylene/plastic sheeting or other material on which well cap and other equipment may be placed.

2.14.5. Procedures

2.14.5.1. Equipment Maintenance

1. The procedures listed below are consistent with Stone SOP SEI-5.1.5.
2. Records will be maintained of all inspection, maintenance, testing, and calibrating operations. These records will contain, at a minimum:
    a. Dates of operation.
    b. Whether maintenance was routine or non-routine.
    c. Whether maintenance followed written SOPs.
3. If non-routine repairs are performed, at a minimum, the following will be documented in addition to the items in 2 above:
    a. Nature of the defect.
    b. How and when the defect was discovered.
    c. Any remedial or correction action taken in response to the defect.
4. Inspection, maintenance, testing and calibration efforts will be documented in the field logbook. The records shall include:
    a. The name of the item of equipment;
    b. The manufacturer’s name, type identification, and serial number or other unique identification;
    c. Date received and date place in service (if available);
    d. Current location; where appropriate;
    e. Condition when received (e.g., new, used, reconditioned);
    f. Copy of the manufacturer's instructions,
    g. Dates and results of calibrations and/or verifications and date of the next calibration and/or verification;
    h. Details of maintenance carried out to date and planned for the future; and,
    i. History of any damage, malfunction, modification or repair
    j. Equipment which is malfunctioning and has been taken out of service, it shall be tagged as such.

2.14.5.2. Equipment Decontamination

1. The procedures listed below are consistent with Stone SOP SEI-5.1.5.

2. The following procedure applies to decontamination of hand-held sampling devices used for collecting samples for trace organic or inorganic constituent:
    a. Clean the device with tap water and soap, using a brush if necessary to remove particulate matter and surface films.
    b. Rinse thoroughly with tap water.
    c. Rinse thoroughly with analyte- or organic-free water.
    d. If necessary and appropriate, rinse thoroughly with solvent. Do not solvent-rinse polyvinyl chloride or plastic items.
    e. Rinse thoroughly with organic/analyte free water, or allow equipment to dry completely.
    f. Remove the equipment from the decontamination area. Equipment stored overnight should be wrapped in aluminum foil and covered with clean, unused plastic.
3. The following procedure is applies to decontamination of heavy equipment to be used for soil borings, etc.:
    a. Wipe/scrape the equipment to remove gross contamination directly into IDW containers.
    b. Thoroughly clean the equipment using high pressure steam.
    c. Rinse thoroughly with tap water.
    d. Remove the equipment from the decontamination area.

The decontamination area will be surrounded by "Caution" tape, and all hazardous materials will be removed at the end of each day, to prevent accidental exposure.

2.15. Management of Investigation-Derived Waste

2.15.1. Site Background

As part of RI/FS Task Number 3, groundwater samples will be collected from approximately 51 monitoring wells at the Site which will generate purge water. As part of RI/FS Task Number 5, soil borings will be advanced and NAPL monitoring wells may be installed in the Source Area, which will generate soil cuttings, and development and purge water. As part of Task Number 6, three shallow overburden monitoring wells will be installed on the North Kingstown DPW Property, which will generate soil cuttings, and development and purge water.. All seven RI/FS tasks will generate some amount of decontamination fluids. Therefore, each of the RI/FS tasks will require managing IDW.

2.15.2. Sampling Objectives

There are four types of IDW will be generated during the RI/FS investigation:

1. Solid waste suitable for disposal at a certified solid waste landfill (non-hazardous);
2. Solid wastes containing hazardous constituents at concentrations above Remediation Regulation standards for residential direct exposure;
3. Liquid waste suitable for discharge to the ground surface; and
4. Liquid waste generated from investigation activities such as decontamination, monitoring well development and sampling.

Management of IDW, described in more detail below, will follow the requirements outlined in RIDEM's Policy Memo 95-01: *Guidelines for the Management of Investigation Derived Wastes* (RIDEM, 1995).

2.15.3. Sample Location and Frequency

Stone will be responsible for the proper management of all IDW, however the USACE (and Navy where appropriate) will be considered the "generator" of any IDW considered hazardous waste and will therefore need to sign any required manifests. The point of contact responsible for IDW management is Stone PM, Joseph Schmidl. IDW will be disposed of within 30 days of completion of that phase of the field investigation site work (including receipt of laboratory analysis).

Any IDW determined to be hazardous will be containerized and labeled in accordance with Resource Conservation and Recovery Act and RIDEM hazardous waste regulations. All non-hazardous waste IDW storage containers will be labeled with the following information: class of IDW; source area; data of generation; and generator name, address and phone number. To the extent possible based on observations and field screening results, IDW will be segregated according to potential level of contamination (i.e., heavily versus lightly contaminated).

2.15.3.1.1. Sampling and Disposal Procedures for Solid Waste

Solid IDW for this program is anticipated to include sampling equipment (e.g., disposable valves, fittings and sample tubing used to collect samples), personal protective equipment (PPE), spent carbon, and soil/cuttings from the installation of wells. The plastic, glass, paper, and PPE IDW will be double-bagged in plastic trash bags and placed into a dumpster for eventual disposal at a lined landfill. Solid soil IDW will be segregated based on its presumed level of contamination, based on background information and observations during the investigation, in order to minimize the amount of solid IDW which will require off-site disposal.

The monitoring well drilling, development, and sampling will create approximately 1 cubic yard of solid IDW. A number of the proposed sampling locations are off the Property. Solid soil IDW from off-Property locations will be collected and moved to the Property. The solid IDW will be temporarily stored in drums or roll-offs on the Property adjacent to Battalion Blvd. Laboratory analysis will be performed to characterize the soil IDW to determine if on- or off-Site disposal is appropriate. Solid IDW generated off the Property (i.e., Hall Creek, North Kingstown DPW, etc.) will be returned to the Property for storage and, if appropriate, on-site disposal. Onsite disposal will be performed only if VOC, SVOC, RCRA metals, PCBs and TPH analytical data all meet RIDEM Remediation Regulation standards for residential direct exposure. A minimum of one characterization sample will be collected. If characterization data indicate RIDEM residential direct exposure standards are not exceeded, the soil IDW will be spread on the ground surface on the Property, immediately southeast of the Source Area, where groundwater flow is southeasterly, away from RI GA Groundwater located to the north of the Property. If RIDEM Remediation Regulation standards for residential direct exposure are exceeded by the soil IDW, the IDW will be containerized, characterized, and ultimately disposed of based on results of characterization sampling for VOCs. The determination will be based on results of analysis once a representative amount of soils has accumulated. A temporary USEPA ID number will be obtained for the Site to be used for the transport and disposal of solid wastes containing contaminants in excess of acceptable levels for onsite disposal. The offsite transport and disposal will be conducted by a qualified contractor in accordance with state and federal regulations.

2.15.3.1.2. Sampling and Disposal Procedures for Liquid Waste

Liquid IDW for this program may include purge water from sampling activities, equipment and field rinse water, and decontamination water. Small amounts of liquid IDW purge water or decontamination water will be dispersed onto the ground surface without treatment near the location of the source of the liquid IDW and in such a manner as to prevent erosion and to prevent the water from entering surface water or storm drains.

Monitoring well drilling, development, and sampling may create hundreds of gallons of liquid IDW. A number of the proposed sampling locations are off the Property. For locations outside the Property (i.e., North Kingstown DPW, etc.), liquid IDW well development water will be containerized and returned to the Property, and temporarily stored in drums on the Property adjacent to Battalion Boulevard. Laboratory analysis will be performed to characterize the liquid IDW to determine if on-Site disposal without treatment is appropriate. On-site disposal without treatment will be performed only if VOC, SVOC, RCRA metals, PCBs and TPH analytical data all meet RIDEM RI GB Groundwater Standards. A minimum of one characterization sample will be collected. If VOCs or other organic constituents exceed RIDEM RI GB Groundwater Standards, the water will be removed from the Property and disposed of in accordance with State and Federal regulations. The discharges of liquid IDW suitable for on-site disposal will be made on the Property, immediately southeast of the Source Area, where groundwater flow is southeasterly, away from RI GA Groundwater located to the north of the Property. Stone will ensure that individual VOC concentrations meet RI GB standards and do not exceed the groundwater concentrations most recently documented in well EA-118S.

2.16. Site Restoration

2.16.1. Site Background

Most of the RI/FS tasks have the potential to impact some portion of the Site. Therefore, each of the RI/FS tasks may require some degree of site restoration.

2.16.2. Sampling Objectives

The objective of this procedure is to restore areas impacted by RI/FS investigation tasks to their original condition following completion of the field work.

Avoidance of site impacts will be the primary means of minimizing adverse impacts. Site restoration will include patching holes in pavement with asphalt, and repair and re-seeding of ruts or damage to maintained vegetation (mowed areas).

2.16.3. Sample Location and Frequency

The following areas will be subject to field activities as part of the RI/FS investigation:

- Twelve pore water/surface water/sediment sample locations along Hall Creek and its vicinity, depicted on Figure 2;
- Approximately 30 soil boring locations and up to three NAPL monitoring wells in the vicinity of well EA-102D, depicted on Figure 3;
- Up to two shallow overburden groundwater monitoring wells at the North Kingstown DPW Property, depicted on Figure 4;
- Up to 51 groundwater monitoring wells across the Site, depicted on Figures 5a and 5b; and
- Up to two residences where well water samples may be collected, depicted on Figure 6.

The Site will be restored immediately following the completion of all field activities in a given area.

2.16.4. Sampling Equipment and Procedures

2.16.4.1. Sampling Equipment

- Digital camera
- Field logbook

2.16.4.2. Sampling Procedure

1. The condition of the area will be photodocumented prior to the initiation of field activities.
2. The potential impacts to the area from RI/FS tasks will be assessed (i.e., soft ground identified) in advance of field activities.
3. Site-specific work plans will be amended to reflect the findings of the assessment (i.e., equipment will be hand carried to avoid driving on soft ground).
4. As necessary, Stone field personnel or subcontractors will repair damage to the Site, in a manner approved by the Stone and USACE Project Managers.
5. The final condition of the repaired impact will be photodocumented.

2.17. Bedrock Description and Measurement

2.17.1. Site Background

A substantial portion of groundwater contamination migrates from the Source Area via bedrock fractures. The orientation and aperture of these fractures strongly influences contaminant migration. These data are important for the evaluation of the nature and extent of contamination in the RI, as well as for the evaluation of remedial alternatives in the FS. These data have previously been collected for bedrock outcrops in the vicinity of Former Nike Battery PR-58, and recent construction of a stormwater detention pond west of the Source Area has resulted in the exposure of a new bedrock outcrop.

2.17.2. Sampling Objectives

The objective of this procedure is to document the type of bedrock present and the orientation of observed fractures in a newly-exposed bedrock outcrop west of the Source Area.

2.17.3. Sample Location and Frequency

Observations and measurements will be made along the length of the bedrock outcrop.

2.17.4. Sampling Equipment and Procedures

2.17.4.1. Sampling Equipment

- Digital camera
- Field logbook
- Brunton® Pocket Transit

- Pocket knife
- Notched Lexan® sheet

2.17.4.2. Sampling Procedure

1. The bedrock outcrop will be photodocumented prior to the initiation of field activities.
2. The type of bedrock, and any bedding or foliation will be measured.
3. Bedrock fractures will be identified and measured for orientation, aperture, and frequency of repetition. Any associated features, such as lineaments, slickensides, offsets, or mineralization, will be recorded, and the type of mineralization will be determined.
4. For fractures which have insufficient surface area for direct placement of the pocket transit / clinometer, a notched Lexan® sheet will be placed over and aligned with the fracture, and the pocket transit / clinometer will be placed against this plane to measure strike and dip.
5. Planar features will be measured for strike, dip, and aperture. Strike is the azimuth of the line formed between the planar feature and the horizontal plane, and will be measured in degrees from true north. Dip is the angle between the planar feature and the horizontal plane, measured perpendicular to the strike, and will be measured in degrees, with a notation regarding which direction the dip occurs.
6. The bedrock data obtained will be added to the database for the site and processed in the same fashion as previous data.

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3. QUALITY ASSURANCE PROJECT PLAN

Per Task 2 of the *PWS*, issued by USACE on November 5, 2010 and subsequently modified on October 31 and December 12, 2011, the QAPP has been prepared in accordance with *Uniform Federal Policy for Quality Assurance Project Plans* (DTIC ADA 427785), dated March 2005. Please refer to Attachment A of this SAP.

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FIGURES

Figure 1: Site Location Map

Figure 2: Proposed Pore Water, Surface Water, and Sediment Sample Locations

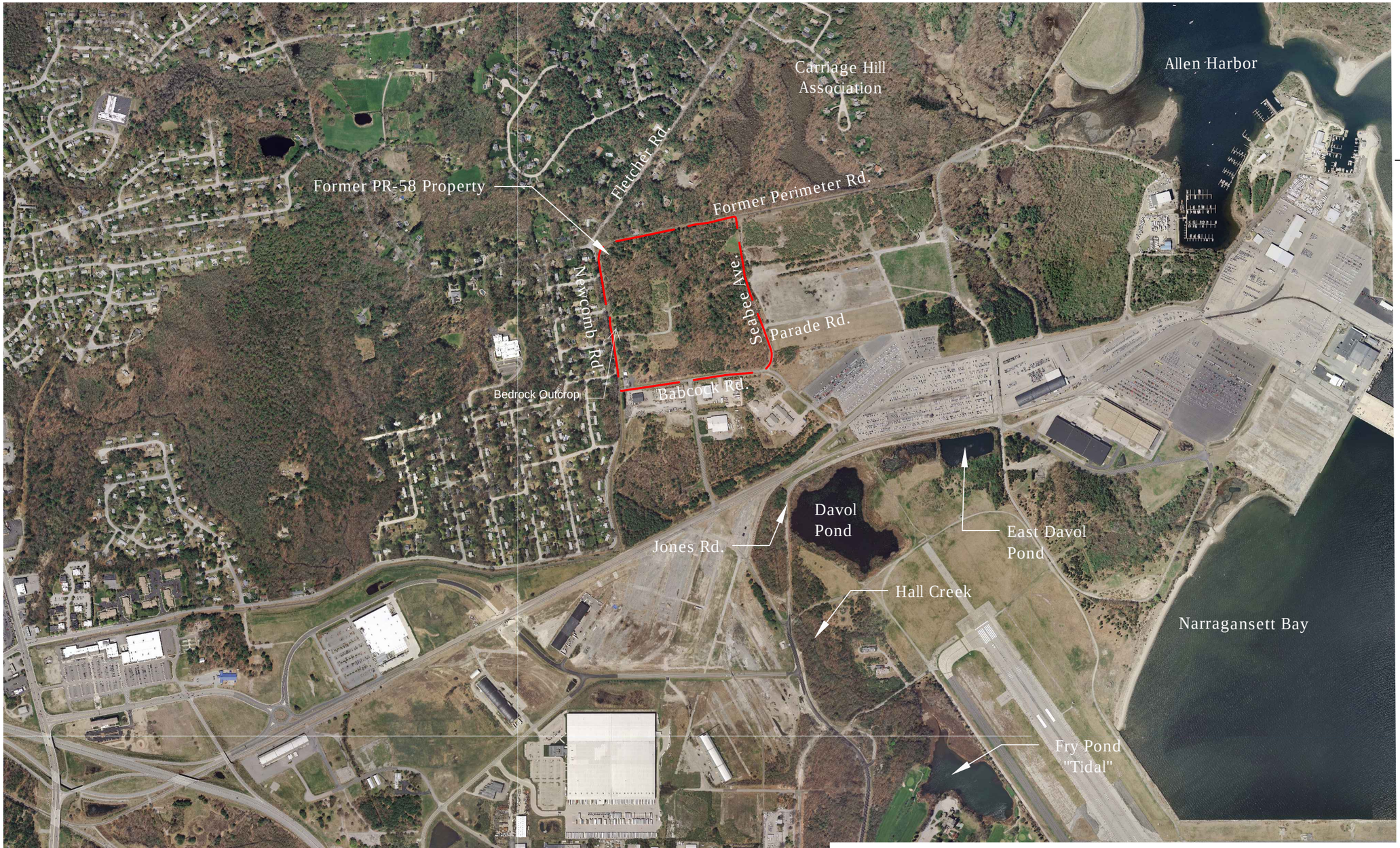
Figure 3: Proposed Source Area Soil Boring Locations

Figure 4: Proposed Well Locations, North Kingstown DPW Property

Figure 5a: Proposed Monitoring Well Sampling Locations - North

Figure 5a: Proposed Monitoring Well Sampling Locations - South

Figure 6: Proposed Residential Supply Well Sampling Locations

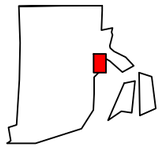


LEGEND

PR-58 Property Boundary

DATUMS

Coord. System: Rhode Island State Plane
Horizontal Datum: feet NAD 1983
Vertical Datum: feet NAVD 1988



Rhode Island

Note:

Source- orthophotography data collection flown April 29 through May 2, 2011. Rhode Island Geographic Information System(RIGIS, 2011).

REVISIONS

#	Date	Drwn	Chk'd	App'd	Description
1	10/26/12	DJL	JS	10/26/12	Displayed 2011 Orthos, Notes updated.

STONE ENVIRONMENTAL INC

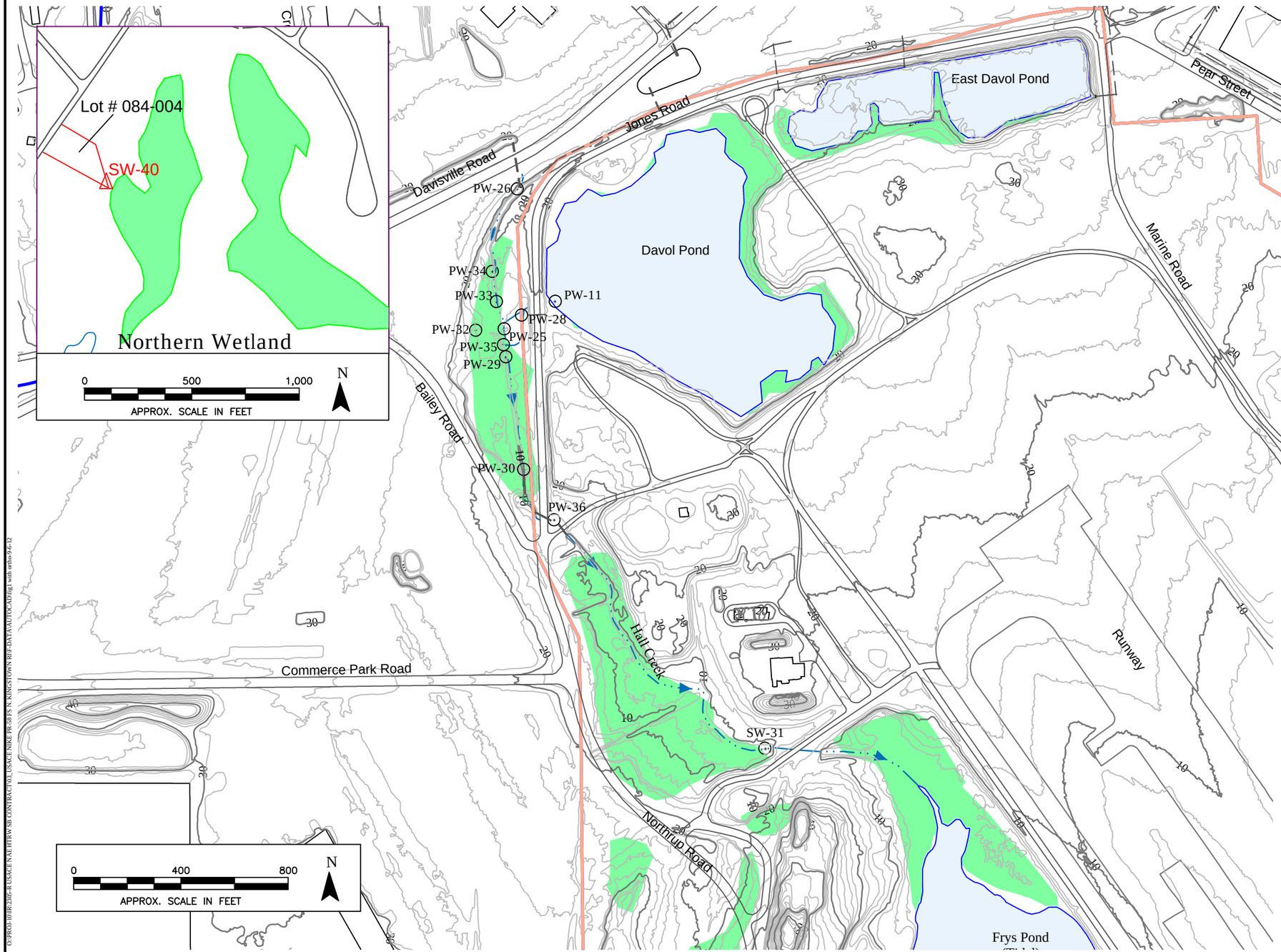
TITLE: Site Location Map

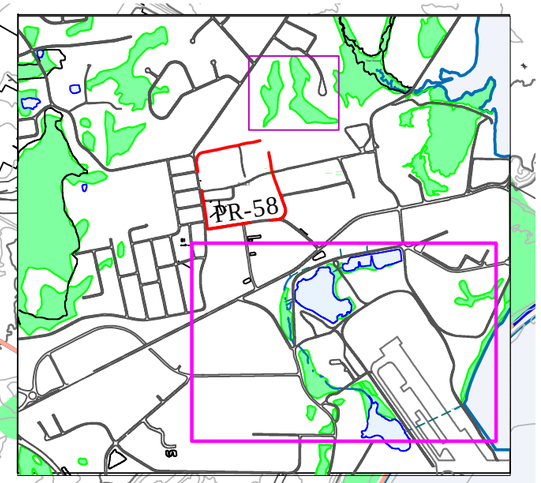
PROJECT: Former Nike Battery PR-58

FIGURE NO.: 1

DATE: 10/26/12 DRAWN BY: DJL CHECKED BY: JS

O:\PROJ-10\R\2305-R USACE NAE HTRW SB CONTRACT\01_USACE NIKE PR-58 FS N, KINGSTOWN RIF-DATA\AUTOCAD





Location Map

Note:

Source- LiDAR terrain data collection flown April 7-8, 2007. Rhode Island formerly used defense sites (sky, 2008).

Survey and GPS data collection performed by the New England District of the United States Army Corps of Engineers as part of the 2007 Synoptic Groundwater Sampling Program.

GPS data collection performed by the USACE NAE - 11/05/08 and 09/24/09

REVISIONS	#	Date	Drwn	Chk'd	App'd	Description
	1	10/27/12	DJL	JS	10/26/12	Displayed Topo, Show Buildings
	2	12/7/12	DJL	JS	12/7/12	Notes updated, Displayed missing wells, updated culvert/drainage.
	3	1/31/13	DJL	JS	1/31/13	darkened 10' contours,increased font, added northern sampling locations
	4	3/18/13	DJL	JS	3/18/13	removed SW-41, and Lot ID

LEGEND

- PW-28 — Multimedia Sample Location
- SW-40 — Surface Water Sample
- Buildings
- Culvert/Drainage
- Stream with Flow Direction
- Wetland
- PR-58 Property Boundary
- Naval Construction Battalion Center Area
- Naval Air Station Area
- Water body
- Map Extent
- Map Inset
- 10 — 10' Ground Surface Contour
- 2' Ground Surface Contour
- Property lines

DATUMS

Coord. System: Rhode Island State Plane
Horizontal Datum: feet NAD 1983
Vertical Datum: feet NAVD 1988

STONE ENVIRONMENTAL INC

TITLE: Pore Water, Surface Water, and Sediment Sample Locations

PROJECT: Former Nike Battery PR-58

FIGURE NO.: 2

DATE: 3/18/13 DRAWN BY: DJL CHECKED BY: JS

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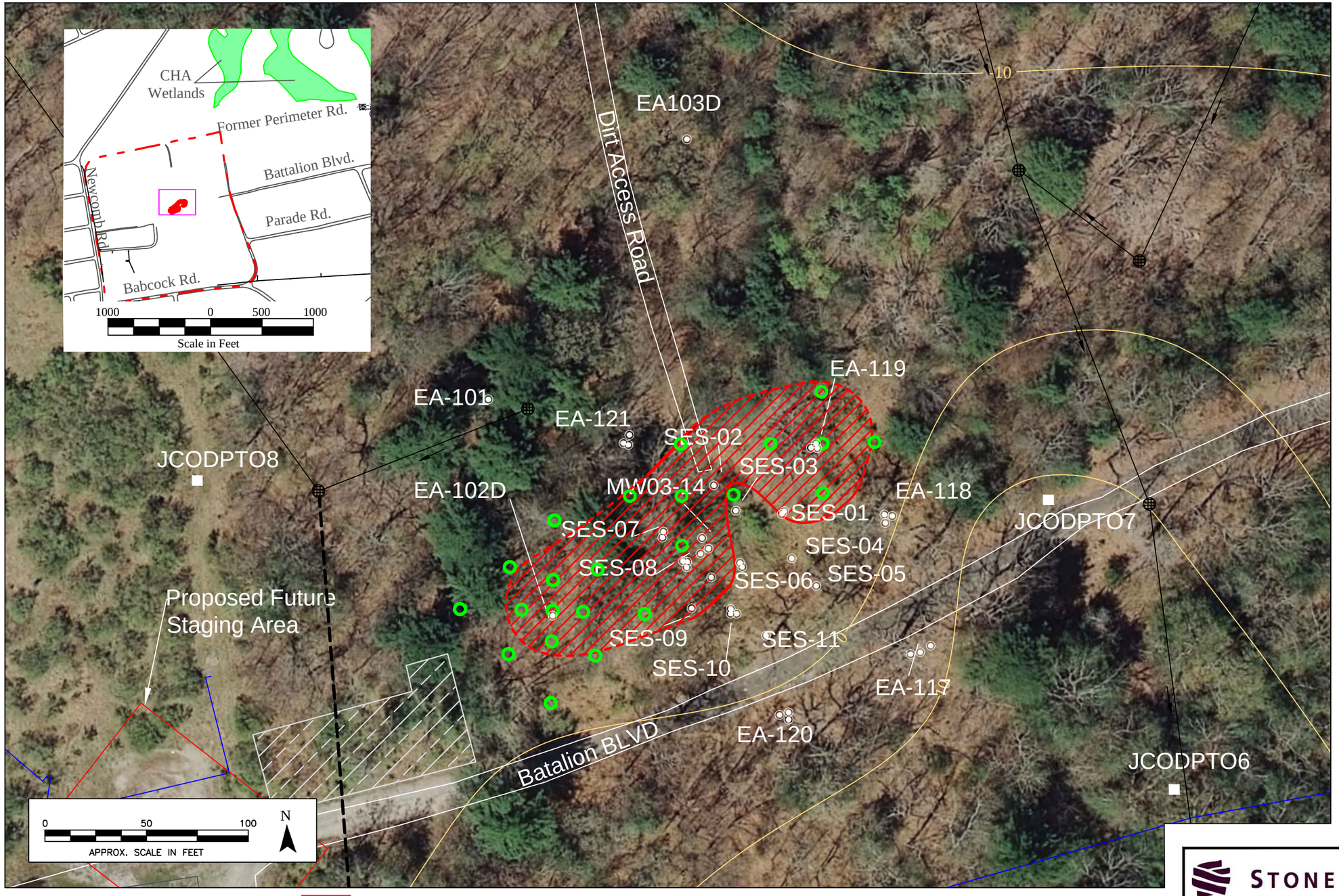


O:\PROJ-10\R2305-R USACE\NAE HTRW SB CONTRACT\05_USACE NIKE PR-58 FS N. KINGSTOWN RI\E-DATA\AUTOCAD\soil borings

LEGEND

- Monitoring Well Location
- Groundwater Profiling Location
- Source Area
- Top of Bedrock Elevation Contours
- Proposed Soil Boring
- Drainage Line
- Map Extent
- Historical Concrete Pad/Slab
- Abandon Water Line
- Unknown Drainage Line
- Catch Basin

Well Key:

"S" Wells are screened across water table or above ISC silty-sand/silt/clay unit.

"D" Wells are screened below ISC silty-sand/silt/clay unit and/or within five feet of the bedrock surface.

"R" Wells are open holes or screened in bedrock.

Notes:

1) Approximate source area location delineated using sample data from groundwater monitoring wells (2009 and 2010 sampling events) screened within deep overburden or bedrock that showed PCA and/or TCE concentrations in excess of 1% of their single-component solubility.

2) Historical soil sampling and groundwater profiling data (2008 event) also were considered in determining the approximate source area location.

Orthophotography data collection flown April 29 through May 2, 2011. Rhode Island Geographic Information System(RIGIS, 2011).

Survey and GPS data collection performed by the New England district of the Unitied States Army Corps of Engineers as part of the 2007 synoptic groundwater sampling program.

GPS data collection performed by the New England District of the Untied States Army Corps of Engineers - 11/5/08 and 9/24/09.

Final closure and post-closure plan for Peabody Clean Industries hazardous waste tank farm (era, 1984).

DATUMS

Coord. System: Rhode Island State Plane
Horizontal Datum: feet NAD 1983
Vertical Datum: feet NAVD 1988

REVISIONS

#	Date	Drwn	Chk'd	App'd	Description
1	10/12 /12	DJL	10/12 /12	JS	Updated orthos, sources, staging area, utilities updated
2	3/13 /13	DJL	3/13 /13	JS	Updated concrete pad location
3	4/26 /13	DJL	4/26 /13	JS	Updated soil boring locations

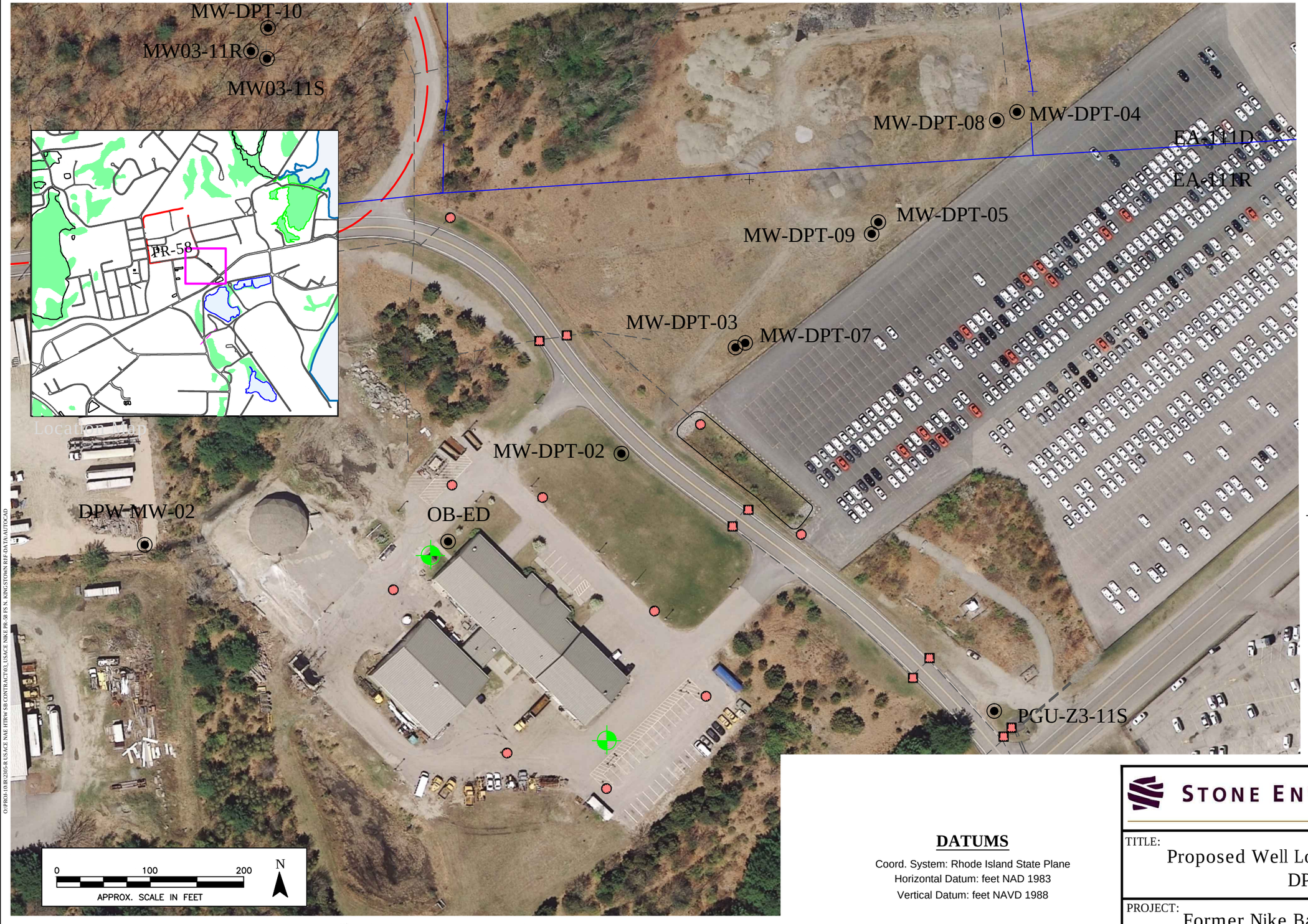
STONE ENVIRONMENTAL INC

TITLE: Proposed Source Area Soil Boring Locations

PROJECT: Former Nike Battery PR-58

FIGURE NO.: 3

DATE: 4/26/13 DRAWN BY: DJL CHECKED BY: JS



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Note:

Source- orthophotography data collection flown April 29 through May 2, 2011. Rhode Island Geographic Information System(RIGIS, 2011).
Survey and GPS data collection performed by the New England District of the United States Army Corps of Engineers as part of the 2007 Synoptic Groundwater Sampling Program.
-GPS data collection performed by the USACE NAE - 11/05/08 and 09/24/09
-Survey by Environmental Planning and Surveying, Inc. - 12/08, 11/09, and 04/10
Data Provided Electronically by the Quonset Point Development Company, Nike Site.dwg and bikepath.dwg, April 2011 (QDC, 2011)

LEGEND

- PR-58 Property Boundary
- Proposed Monitoring Wells
- Existing Monitoring Wells
- Map Extent
- Round Catch Basin
- Grated Catch Basin
- Drain Line
- Water Line

DATUMS

Coord. System: Rhode Island State Plane
Horizontal Datum: feet NAD 1983
Vertical Datum: feet NAVD 1988

REVISIONS

#	Date	Drwn	Chk'd	App'd	Description
1	10/30/12	DJL	JS	10/30/12	Displayed 2011, DPT wells shown. Orthos, Notes-sources updated, drainage.

STONE ENVIRONMENTAL INC

TITLE: Proposed Well Locations, North Kingstown DPW Property

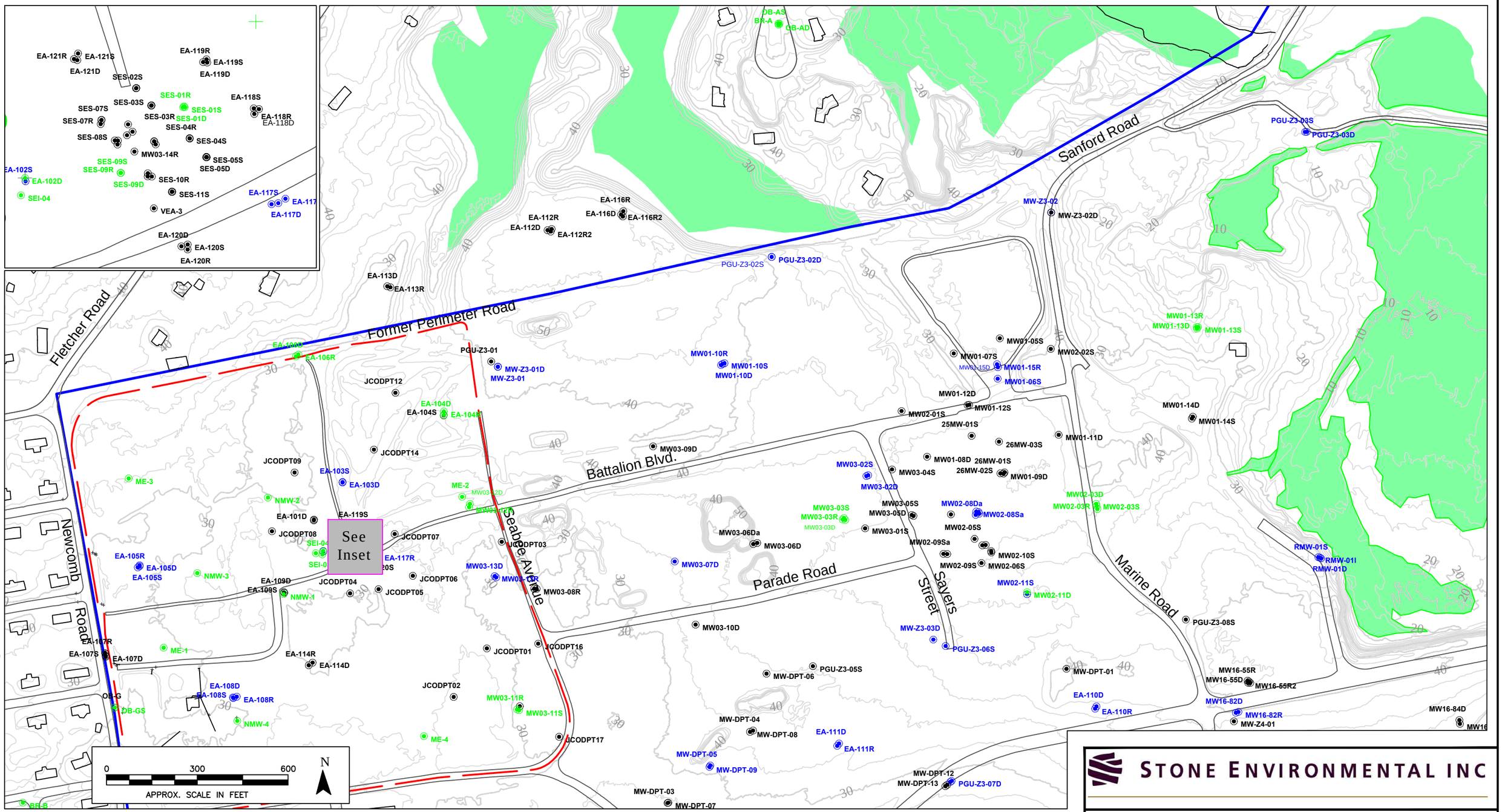
PROJECT: Former Nike Battery PR-58

FIGURE NO.: 4

DATE: 12/14/12

DRAWN BY: DJL

CHECKED BY: JS



LEGEND

- MW03-11R — Proposed Monitoring Wells to be Sampled
- MW03-11R — Proposed Monitoring Wells to Gauged
- EA-108D — Monitoring Wells
- PR-58 Property Boundary
- Wetlands
- Buildings
- Naval Construction Battalion Center Area
- GA/GB Groundwater Classification Boundary
- 10 — 10' Ground Surface Contour
- 2' Ground Surface Contour

DATUMS

Coord. System: Rhode Island State Plane
Horizontal Datum: feet NAD 1983
Vertical Datum: feet NAVD 1988

Notes:

Elevation Source: LiDAR terrain data collection flown April 7-8, 2007. Rhode Island formerly used defense sites (sky, 2008). Survey and GPS data collection performed by the New England District of the United States Army Corps of Engineers as part of the 2007 Synoptic Groundwater Sampling Program.
-GPS data collection performed by the USACE NAE - 11/05/08 and 09/24/09
-Survey by Environmental Planning and Surveying, Inc. - 12/08, 11/09, and 04/10
Data Provided Electronically by the Quonset Point Development Company, Nike Site.dwg and bikepath.dwg, April 2011 (QDC, 2011)

REVISIONS

#	Date	Drwn	Chk'd	App'd	Description
1	10/26/12	DJL	JS	10/26/12	Fixed Boundary, Show Buildings, Notes updated.
2	1/31/13	DJL	JS	1/31/13	darkened 10' contours, increased font size
3	3/13/13	DJL	JS	3/13/13	Changed Wells
4	9/13/13	SAH	JS	9/19/13	Identified gauged well, changed tables, legend, layout

STONE ENVIRONMENTAL INC

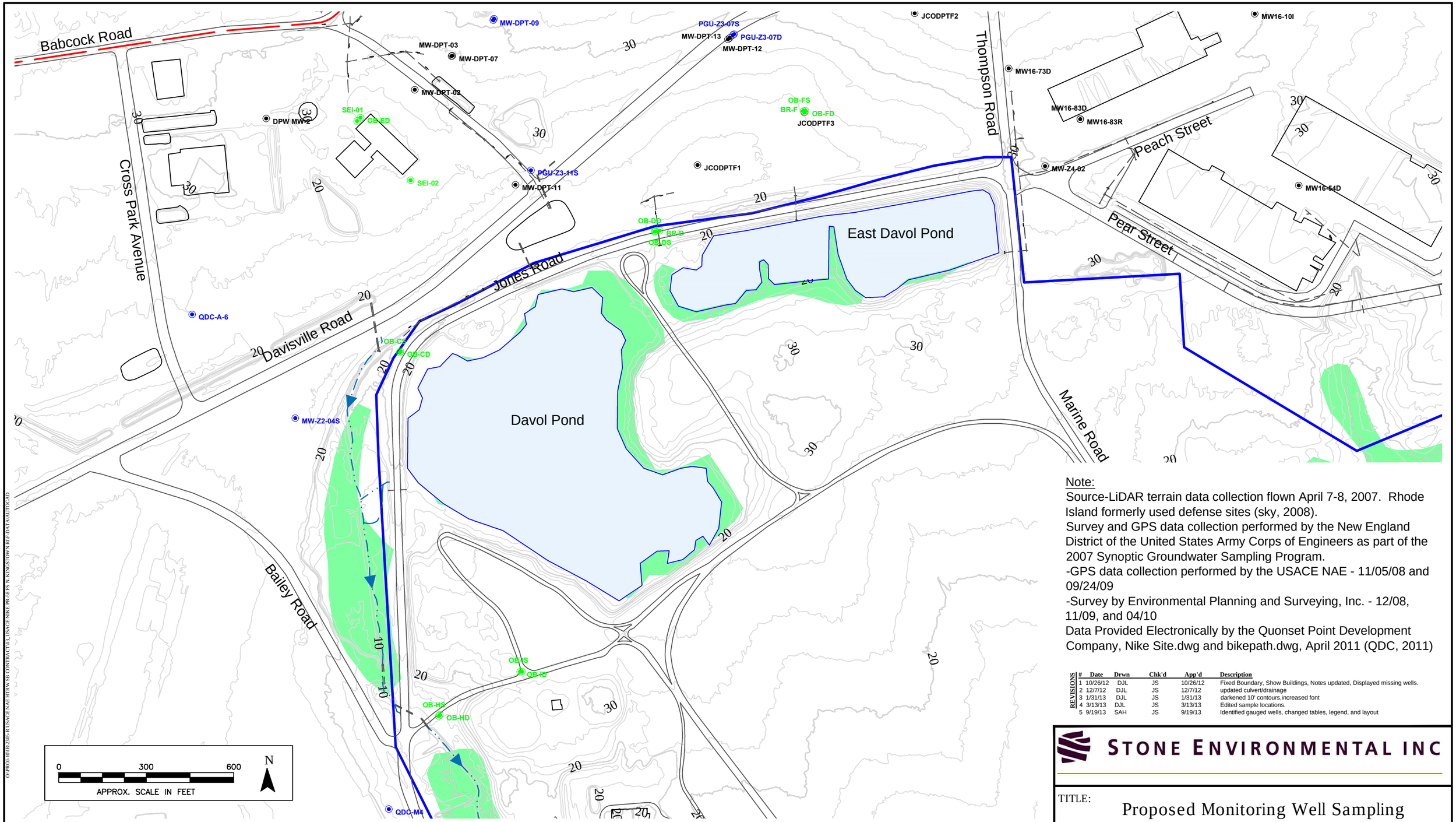
TITLE: Proposed Monitoring Well Sampling Locations - North

PROJECT: Former Nike Battery PR-58

FIGURE NO.: 5a

DATE: 9/19/13 DRAWN BY: SAH CHECKED BY: JS

O:\PROJ\10\R\230\R-USACE\NAE\FRW SB CONTRACT\03_USACE NIKE PR-58 FS 3V, KINGSTOWN RIF-DATA\AUTOCAD



LEGEND

- MW03-11R — Proposed Monitoring Wells to be Sampled
- MW03-11R — Proposed Monitoring Wells to be Gauged
- EA-108D — Monitoring Wells
- PR-58 Property Boundary
- Wetland
- Buildings
- Naval Construction Battalion Center Area
- Water body
- Culvert/Drainage
- 10 — 10' Ground Surface Contour
- 2' Ground Surface Contour

DATUMS

Coord. System: Rhode Island State Plane
Horizontal Datum: feet NAD 1983
Vertical Datum: feet NAVD 1988

Note:
Source-LiDAR terrain data collection flown April 7-8, 2007. Rhode Island formerly used defense sites (sky, 2008).
Survey and GPS data collection performed by the New England District of the United States Army Corps of Engineers as part of the 2007 Synoptic Groundwater Sampling Program.
-GPS data collection performed by the USACE NAE - 11/05/08 and 09/24/09
-Survey by Environmental Planning and Surveying, Inc. - 12/08, 11/09, and 04/10
Data Provided Electronically by the Quonset Point Development Company, Nike Site.dwg and bikepath.dwg, April 2011 (QDC, 2011)

REVISIONS

#	Date	Drwn	Chk'd	App'd	Description
1	10/26/12	DJL	JS	10/26/12	Fixed Boundary, Show Buildings, Notes updated, Displayed missing wells.
2	12/7/12	DJL	JS	12/7/12	updated culvert/drainage
3	1/31/13	DJL	JS	1/31/13	darkened 10' contours,increased font
4	3/13/13	DJL	JS	3/13/13	Edited sample locations.
5	9/19/13	SAH	JS	9/19/13	Identified gauged wells, changed tables, legend, and layout

STONE ENVIRONMENTAL INC

TITLE: Proposed Monitoring Well Sampling Locations - South

PROJECT: Former Nike Battery PR-58

FIGURE NO.: 5b

DATE: 9/19/13 DRAWN BY: SAH CHECKED BY: JS

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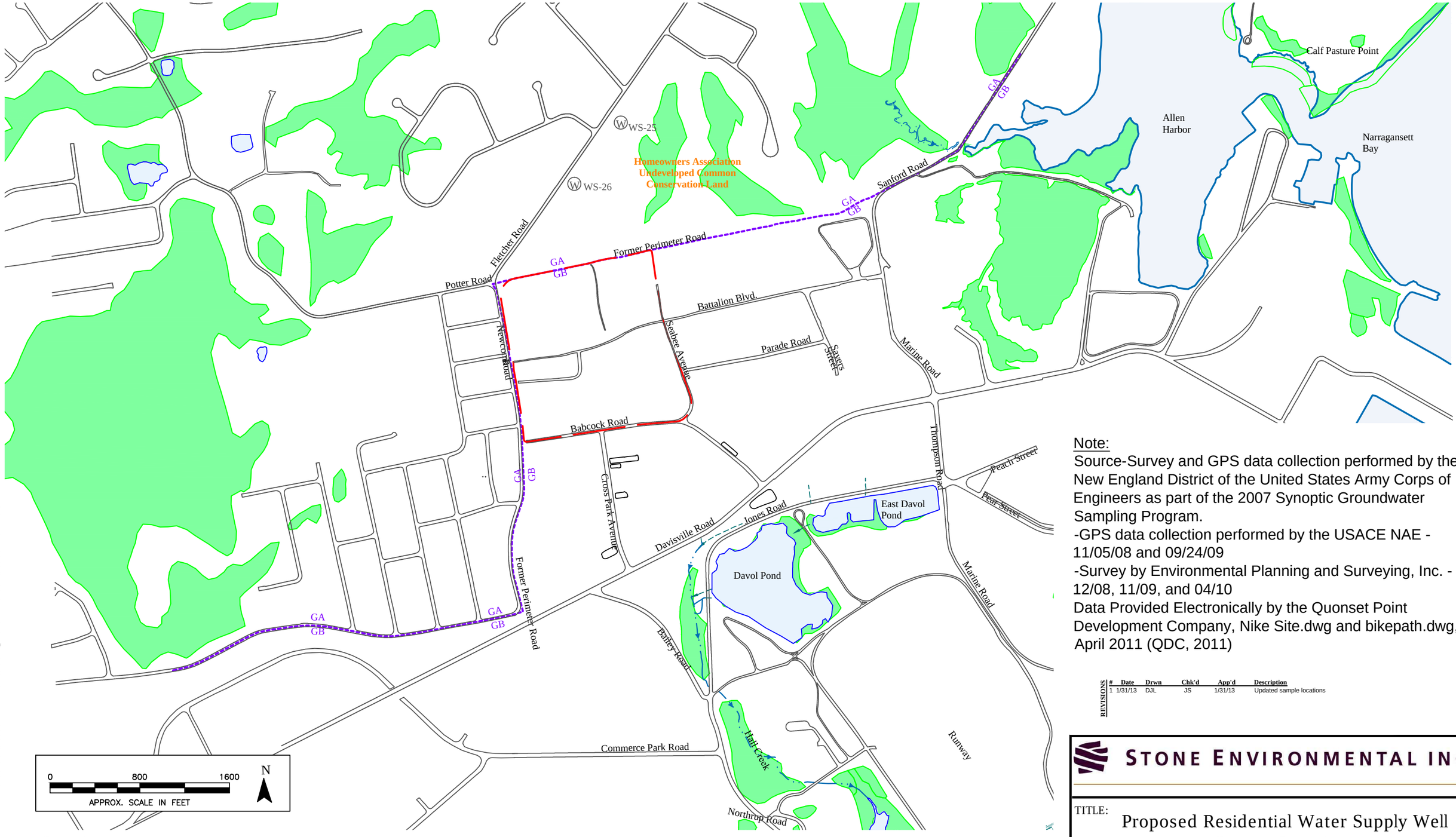

LEGEND

- PR-58 Property Boundary
- Residential Water Supply Area
- WS-25 Residential Wells
- GA/GB Groundwater Classification Boundary
- Culvert
- Stream with Flow Direction
- Buildings
- Wetland
- Water body

DATUMS

Coord. System: Rhode Island State Plane
Horizontal Datum: feet NAD 1983
Vertical Datum: feet NAVD 1988

Note:

Source-Survey and GPS data collection performed by the New England District of the United States Army Corps of Engineers as part of the 2007 Synoptic Groundwater Sampling Program.
-GPS data collection performed by the USACE NAE - 11/05/08 and 09/24/09
-Survey by Environmental Planning and Surveying, Inc. - 12/08, 11/09, and 04/10
Data Provided Electronically by the Quonset Point Development Company, Nike Site.dwg and bikepath.dwg, April 2011 (QDC, 2011)

REVISIONS

#	Date	Drwn	Chk'd	App'd	Description
1	1/31/13	DJL	JS	1/31/13	Updated sample locations

STONE ENVIRONMENTAL INC

TITLE: Proposed Residential Water Supply Well Sample Locations

PROJECT: Former Nike Battery PR-58

FIGURE NO.: 6

DATE: 1/31/13 DRAWN BY: DJL CHECKED BY: JS

O:\PROJ-TOUR\2265-RI\USACE NAE\HTRW SB CONTRACT\02-USACE NIKE PR-58 FS 'N KINGSTOWN RI\F-DATA\AUTOCAD

ATTACHMENTS

A: QUALITY ASSURANCE PROJECT PLAN

QUALITY ASSURANCE PROJECT PLAN

REVISION 2

FORMER NIKE BATTERY PR-58
NORTH KINGSTOWN, RHODE ISLAND
REMEDIAL INVESTIGATION / FEASIBILITY STUDY

FUDS Project Number: D01RI0029 05
CENAE Project Number: 108346

Contract Number: W912WJ-11-D-0001
Stone Project ID: 102305-R-03
October 2013

Prepared for:

US Army Corps of Engineers, New England District
696 Virginia Road
Concord, MA 01742-2751

Prepared by:

Stone Environmental, Inc.
535 Stone Cutters Way
Montpelier, VT 05602
Tel. / 802.229.4541
E-Mail / sei@stone-env.com

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ATTACHMENT A

LIST OF METHOD PERFORMANCE CRITERIA FOR ALL ANALYTES

QAPP WORKSHEET #1: TITLE AND APPROVAL PAGE

Site Name/Project Name: Nike Battery Site PR-58

Site Location: North Kingstown, Rhode Island

Document Title: QAPP-Nike Battery Site PR-58 Remedial Investigation

Lead Organization: U.S. Army Corps of Engineers, New England District (NAE)

Preparer's Name and Organizational Affiliation: Joseph Schmidl and Kim Watson, Stone Environmental, Inc.

Preparer's Address, Telephone Number, and E-mail Address:

535 Stone Cutters Way Montpelier, Vermont 05602 (802) 229-2196 kwatson@stone-env.com

Preparation Date (Day/Month/Year): 06/12/2013

Investigative Organization's Program Manager/Date: _____
Signature

Printed Name/Organization: Seth Pitkin, Stone Environmental, Inc.

Investigative Organization's Project Manager/Date: _____
Signature

Printed Name/Organization: Joseph Schmidl, P.G., C.W.S., Stone Environmental, Inc.

Investigative Organization's Project QA Officer/Date: _____
Signature

Printed Name/Organization: Kim Watson, Stone Environmental, Inc.

Investigative Organization's Project Chemist/Date: _____
Signature

Printed Name/Organization: Michael Rossi, Stone Environmental, Inc.

Lead Organization's Project Manager/Date: _____
Signature

Printed Name/Organization: Casey Haskell, P.G., USACE New England District (NAE)

Concurrence Signatures/Date: _____
Signature

Printed Name/Title: Richard Gottlieb, P.E., Rhode Island Department of Environmental Management

Approval Authority:

Other Approval Signatures/Date: _____
Signature

Printed Name/Title:

Document Control Numbering System: PR58UFPQAPPv2.0.docx

QAPP WORKSHEET #2: QAPP IDENTIFYING INFORMATION

Site Name/Project Name: Former Nike Battery PR-58
Site Location: North Kingstown, Rhode Island
Site Number/Code: D01RI0029 05
Operable Unit:
Contractor Name: Stone Environmental, Inc.
Contractor Number: W912WJ-11-D-0001

Title: Quality Assurance Project Plan
Revision Number: 1
Revision Date: 12 June 2013

Contract Title: Delivery Order 0003
Work Assignment Number: 108346

1. Identify regulatory program: Comprehensive Environmental Response, Compensation and Liability Act (USEPA)
2. Identify concurrence entity: Richard Gottlieb, P. E., Office of Waste Management
3. The QAPP is (select one): ☐Generic ☒Project Specific
4. List dates of scoping sessions that were held: 6 March, 16 August, and 1 November 2012
5. List dates and titles of QAPP documents written for previous site work, if applicable:

Title	Approval Date

6. List organizational partners (stakeholders) and connection with lead organization:
   Jeff Dale. Remedial Project Manager, BRAC PMO Northeast
   Christine A.P. Williams, Federal Facility Superfund Section, US EPA New England
   Steve King, Quonset Development Corp.
   David Barney, BRAC Environmental Coordinator, NAS South Weymouth
   Susan Licardi, Town of North Kingstown
7. List data users:
   Stone Environmental, Inc.
   USACE
   RIDEM
   USEPA
   US Navy, BRAC
8. If any required QAPP elements and required information are not applicable to the project, then circle the omitted QAPP elements and required information on the attached table. Provide an explanation for their exclusions below:

QAPP Worksheet #2 (continued)
QAPP Identifying Information

QAPP elements and required information that are not applicable to the project are circled and an explanation is provided in the QAPP.

Required QAPP Element(s) and Corresponding QAPP Section(s)	Required Information	QAPP Worksheet # or Crosswalk to Related Document
Project Management and Objectives		
2.1 Title and Approval Page	- Title and Approval Page	1
2.2 Document Format and Table of Contents 2.2.1 Document Control Format 2.2.2 Document Control Numbering System 2.2.3 Table of Contents 2.2.4 QAPP Identifying Information	- Table of Contents - QAPP Identifying Information	 2
2.3 Distribution List and Project Personnel Sign-Off Sheet 2.3.1 Distribution List 2.3.2 Project Personnel Sign-Off Sheet	- Distribution List - Project Personnel Sign-Off Sheet	3 4
2.4 Project Organization 2.4.1 Project Organizational Chart 2.4.2 Communication Pathways 2.4.3 Personnel Responsibilities and Qualifications Table 2.4.4 Special Training Requirements and Certification	- Project Organizational Chart - Communication Pathways - Personnel Responsibilities and Qualifications Table - Special Personnel Training Requirements Table	5 6 7 8
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2.6 Project Quality Objectives and Measurement Performance Criteria 2.6.1 Development of Project Quality Objectives Using the Systematic Planning Process 2.6.2 Measurement Performance Criteria	- Site Specific PQOs - Measurement Performance Criteria	11 12
2.7 Secondary Data Evaluation	- Sources of Secondary Data and Information - Secondary Data Criteria and Limitations Table	 13

Required QAPP Element(s) and Corresponding QAPP Section(s)	Required Information	QAPP Worksheet # or Crosswalk to Related Document
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Measurement/Data Acquisition		
3.1 Sampling Tasks 3.1.1 Sampling Process Design and Rationale 3.1.2 Sampling Procedures and Requirements 3.1.2.1 Sampling Collection Procedures 3.1.2.2 Sample Containers, Volume, and Preservation 3.1.2.3 Equipment/Sample Containers Cleaning and Decontamination Procedures 3.1.2.4 Field Equipment Calibration, Maintenance, Testing, and Inspection Procedures 3.1.2.5 Supply Inspection and Acceptance Procedures 3.1.2.6 Field Documentation Procedures	- Sampling Design and Rationale - Sample Location Map - Sampling Locations and Methods/SOP Requirements Table - Analytical Methods/SOP Requirements Table - Field Quality Control Sample Summary Table - Sampling SOPs - Project Sampling SOP References Table - Field Equipment Calibration, Maintenance, Testing, and Inspection Table	17 18 19 20 21 22
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Required QAPP Element(s) and Corresponding QAPP Section(s)	Required Information	QAPP Worksheet # or Crosswalk to Related Document
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4.2 QA Management Reports	- QA Management Reports Table	33
4.3 Final Project Report		
Data Review		
5.1 Overview		
5.2 Data Review Steps 5.2.1 Step I: Verification 5.2.2 Step II: Validation 5.2.2.1 Step IIa: Validation Activities 5.2.2.2 Step IIb: Validation Activities 5.2.3 Step III: Usability Assessment 5.2.3.1 Data Limitations and Actions from Usability Assessment 5.2.3.2 Activities	- Verification (Step I) Process Table - Validation (Steps IIa and IIb) Process Table - Validation (Steps IIa and IIb) Summary Table - Usability Assessment	34 35 36 37
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QAPP WORKSHEET #3: DISTRIBUTION LIST

QAPP Recipients	Title	Organization	Telephone Number	Fax Number	E-mail Address	Document Control Number
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David Lubianez	Project Chemist	NAE	978-318-8311		David.J.Lubianez@usace.army.mil	v2.0
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Cynthia Colquitt	Human Health Risk Assessor	NAE	978-318-8042		Cynthia.A.Hanna@usace.army.mil	v2.0
Michael Penko	Environmental Risk Assessor	NAE	978-318-8139		Michael.Penko@usace.army.mil	v2.0
Richard Gottlieb, P.E.	Project Manager	RIDEM	401-222-2797		richard.gottlieb@dem.ri.gov	v2.0
David Barney	Project Manager	US Navy	617-753-4656		david.a.barney@navy.mil	v2.0
Christine Williams	Project Manager	US EPA	617-918-1384	617-918-1291	williams.christine@epa.gov	v2.0
Joseph Schmidl, P.G., C.W.S.	Project Manager	Stone Environmental, Inc.	802-229-5377	802-229-5417	JSchmidl@Stone-Env.com	v2.0
Seth Pitkin	Senior Hydrogeologist/Vice President	Stone Environmental, Inc.	802-229-2192	802-229-5417	spitkin@stone-env.com	v2.0
Lee Rosberg	Field Operations Leader/Site Safety Officer	Stone Environmental, Inc.	802-229-6434	802-229-5378	lrosberg@stone-env.com	v2.0
Kim Watson	QA Officer/Health & Safety Officer	Stone Environmental, Inc.	802-229-2196	802-229-5417	kwatson@stone-env.com	v2.0
Michael Rossi	Senior Chemist/Laboratory Manager	Stone Environmental, Inc.	802-229-2194	802-229-5417	mrossi@stone-env.com	v2.0

QAPP Worksheet #3 continued
Distribution List

QAPP Recipients	Title	Organization	Telephone Number	Fax Number	E-mail Address	Document Control Number
Larry Pannell, P.G., P.M.P.	Project Manager	Watermark	978-452-9696	978-453-9988	lawrence.pannell@watermarkenv.com	v2.0
Jeffrey Reid, P.G.	Project Manager	Hager-Richter Geoscience	603-893-9944	603-893-8313	jeff.reid@hager-richter.com	v2.0
Michael Murphy	Risk Assessor	AMEC	978-692- 5365	978-692-6633	michael.murphy@amec.com	v2.0
Tony Rodolakis	Department Manager	AMEC	978-692- 5365	978-692-6633	tony.rodolakis@amec.com	v2.0
Laurel Stoddard	Laboratory Manager	ESS Laboratory (ESS)	401-461-7181 ext 3066	401-461-4486	lstoddard@thielsch.com	v2.0
Jennifer Ericson	Laboratory Manager	Microbial Insights	865-573-8188	865-573-8133	info@microbe.com	v2.0
Hugh Peek	Laboratory Manager	Core Laboratories	978-635-0424	978-635-0266	Hugh.Peek@corelab.com	v2.0

QAPP WORKSHEET #4: PROJECT PERSONNEL SIGN-OFF SHEET

Organization: U.S. Army Corps of Engineers New England District (NAE)

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
Casey Haskell, P.E.	Project Manager	978-318-8398		
Tracy Dorgan, P.G.	Geologist	978-318-8499		
David Lubianez	Project Chemist	978-318-8311		
Dan Groher, P.E.	Innovative Technologies	978-318-8404		
Sheila Harvey	Health & Safety Officer	978-318-8504		
Cynthia Hanna	Risk Assessor	978-318-8042		
Michael Penko	Risk Assessor	978-318-8139		

Organization: Stone Environmental, Inc.

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
Joseph Schmidl, P.G., C.W.S.	Project Manager	802-229-5377		
Seth Pitkin	Senior Hydrogeologist/Vice President	802-229-2192		
Lee Rosberg	Field Operations Leader/Site Safety Officer	802-229-5378		
Kim Watson	QA Officer/Health & Safety Officer	802-229-2196		
Michael Rossi	Senior Chemist/Laboratory Manager	802-229-2194		

Organization: Rhode Island Department of Environmental Management

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
Richard Gottlieb, P.E.	Project Manager	401-222-2797		

Organization: Watermark

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
Larry Pannell, PG, PMP	Project Manager	978-452-9696		

QAPP Worksheet #4: Project Personnel Sign-Off Sheet (Concluded)

Organization: Hager-Richter Geoscience

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
Jeff Reid, P.G.	Senior Geophysicist	603-893-9944		

Organization: AMEC

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
Michael Murphy	Risk Assessor	978-392- 5365		
Tony Rodolakis	Risk Assessor	978-392- 5365		

Organization: ESS Laboratory

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
Laurel Stoddard	Laboratory Manager	401-461-7181x3066		

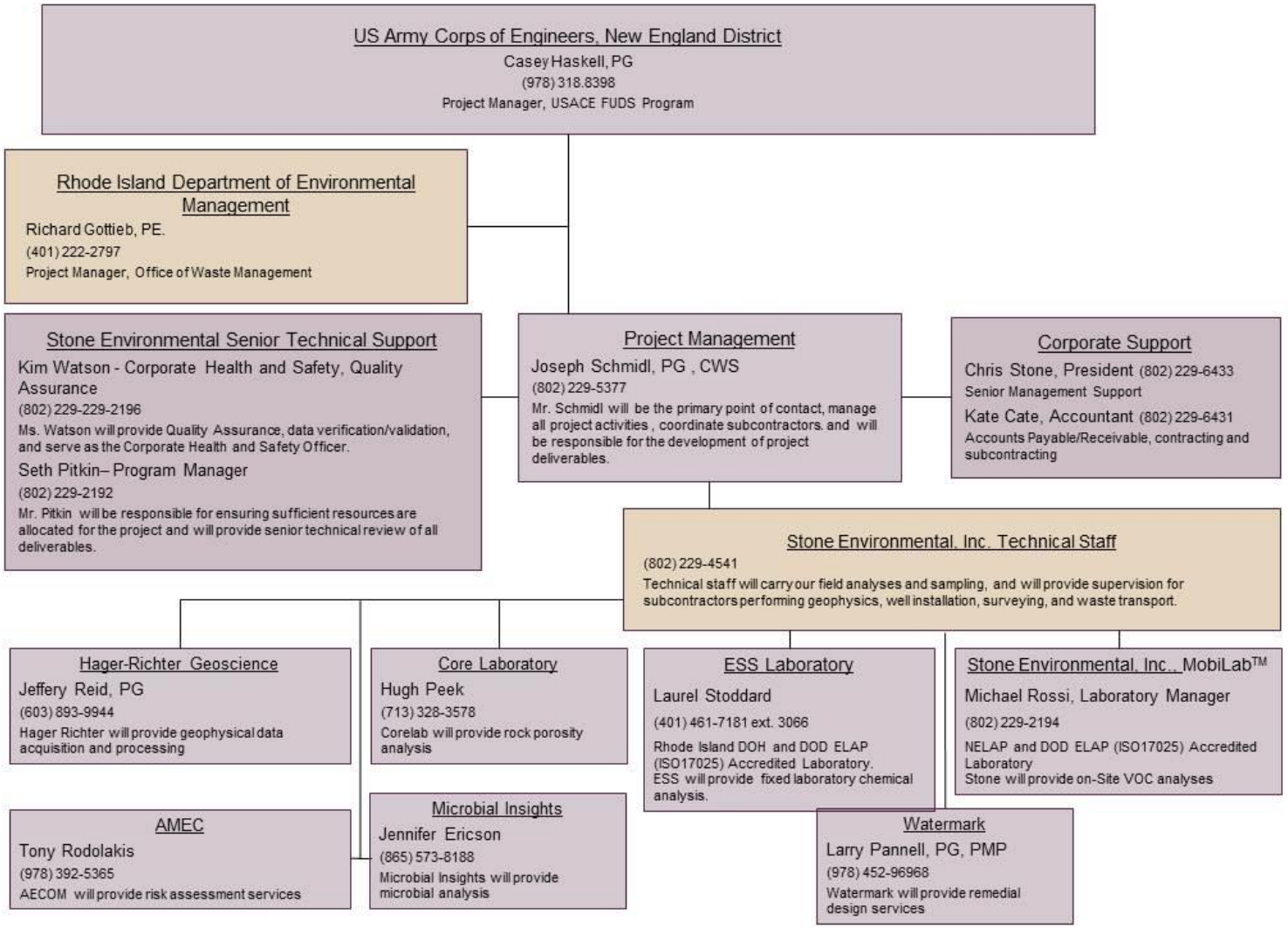
Organization: Microbial Insights

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
Jennifer Ericson	Laboratory Manager	865-573-8188		

Organization: Core Laboratory

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
Hugh Peek	Laboratory Manager	978-635-0424		

QAPP WORKSHEET #5: PROJECT ORGANIZATIONAL CHART



QAPP WORKSHEET #6: COMMUNICATION PATHWAYS

☐ Worksheet Not Applicable (State Reason)

Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (Timing, Pathways, Etc.)
Point of Contact with NAE	Contractor Project Managers	Seth Pitkin, Joseph Schmidl, P.G	802-229-2192 802-229-5377	All materials and information about the project will be forwarded to Casey Haskell, P.G.
Manage all Project Phases	Contractor Project Managers	Joseph Schmidl, P.G	802-229-5377	Notify Casey Haskell, P.G. of field-related problems by COB the next business day
QAPP changes	QAPP Preparer	Kim Watson and Joseph Schmidl	802-229-2196	Will notify about minor changes to the QAPP; prepare QAPP amendments for major changes.
QAPP Amendments	Lead Organization's Project Chemist	David Lubianez	978-318-8021	Any major changes to the QAPP must be approved by David Lubianez before the changes can be implemented.
Daily Field Progress Reports	Sampling Team Leader	Joseph Schmidl, P.G	802-229-5377	Will provide verbal daily field progress reports to Casey Haskell, P.G.
Weekly Field Progress Reports	Field Work Manager	Joseph Schmidl, P.G	802-229-5377	Will provide written reports of weekly field progress to Casey Haskell, P.G.
Recommendations to stop work and initiate corrective action	Field Work Manager	Joseph Schmidl, P.G	802-229-5377	Will notify Casey Haskell, P.G. where unforeseen circumstances require the stoppage of work and/or immediate remedial actions.
Reporting Lab Data Quality Issues	Laboratory Quality Assurance Officer	Laurel Stoddard (fixed) Michael Rossi (mobile)	401-461-7181x3066 802-229-2194	All QA/QC issues with project field samples will be reported to the Filed Team Leader by the laboratory QAOs.
Field and Analytical Corrective Actions	Contractor Quality Assurance Officer	Kim Watson	802-229-2196	The need for corrective action for field and analytical issues will be determined.
Release of Analytical Data	Contractor Quality Assurance Officer	Kim Watson	802-229-2196	No analytical data can be released until validation is completed and has approved the release.

QAPP WORKSHEET #7: PERSONNEL RESPONSIBILITIES AND QUALIFICATIONS

☐ Worksheet Not Applicable (State Reason)

Name	Title	Organizational Affiliation	Responsibilities	Education and Experience Qualifications
Casey Haskell, P.G.	Lead Organization's Project Manager	NAE	Oversees project	
Seth Pitkin	Contractor Project Officer	Stone Environmental, Inc.	Manages project, main point of contact with NAE	M.S., Hydrogeology, over 25 yrs experience.
Joseph Schmidl, P.G.	Contractor Project Manager	Stone Environmental, Inc.	Manages project, point of contact with NAE, technical hydrogeologic interpretation and review	M.S., P.G., Geology, over 22 yrs experience.
Kim Watson	Contractor QA Officer and H&S Officer	Stone Environmental, Inc.	QA Oversight and Oversees HS for field activities	RQAP-GLP B.S., Environmental Engineering Technology, 31 yrs. experience.
Lee Rosberg	Contractor Field Operations Leader, Site Safety Officer	Stone Environmental, Inc.	Data Collection, Site Health and Safety	B.S. Geology, over 6 yrs experience.
Michael Murphy	Risk Assessor	AECOM	Performs human health risk assessment	M.S., Public Health, 30 yrs experience
Tony Rodolakis	Risk Assessor	AECOM	Performs ecological risk assessment	M.ESc., 18 yrs experience

QAPP WORKSHEET #8: SPECIAL PERSONNEL TRAINING REQUIREMENTS TABLE

☐ Worksheet Not Applicable (State Reason)

No additional specialized training will be required.

Project Function	Specialized Training- Title or Description of Course	Training Provider	Training Date	Personnel/Groups Receiving Training	Personnel Titles/Organizational Affiliation	Location of Training Records/Certificates
Site Workers	OSHA 1910.120	Worker Specific	Worker-Specific training within the past 14 months	Stone Employees	Seth Pitkin, Project Officer, Stone Joseph Schmidl, P.G., Project Manager, Stone Kim Watson, QA/HSO, Stone	Stone Environmental, Inc. 535 Stone Cutters Way, Montpelier, VT 05602

QAPP WORKSHEET #9: PROJECT PLANNING DOCUMENTATION

☐ Worksheet Not Applicable (State Reason)

Project Scoping Session Participants Sheet

Project Name: Nike Battery PR-58 Investigation **Projected Date(s) of Sampling:** May 2013 **Project Manager:** Joseph Schmidl	**Site Name:** Nike Battery PR-58 Site **Site Location:** North Kingstown, RI

Date of Session: 7 November 2012
Scoping Session Purpose: Finalize Scope of Work

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Casey Haskell, P.E.	Project Manager	CENAE	978-318-8398	Casey.Haskell@usace.army.mil	Project Manager
Tracy Dorgan, P.G.	Geologist	CENAE	978-318-8499	Tracy.H.Dorgan@usace.army.mil	Geologist
David Lubianez	Project Chemist	CENAE	978-318-8311	David.J.Lubianez@usace.army.mil	Project Chemist
Dan Groher, P.E.	Project Engineer	CENAE	978-318-8404	Daniel.M.Groher@usace.army.mil	Innovative Technologies
Cynthia Colquitt	Risk Assessor	CENAE	978-318-8042	Cynthia.A.Hanna@usace.army.mil	Human Health Risk Assessor
Michael Penko	Risk Assessor	CENAE	978-318-8139	Michael.Penko@usace.army.mil	Environmental Risk Assessor
Joseph Schmidl, P.G., C.W.S	Project Manager	Stone	802-229-5377	JSchmidl@Stone-Env.com	Project Manager

Comments/Decisions: Reviewed Client Draft Work Plan. Evaluated potential data gaps. Requested that Stone further investigate certain data gaps and resubmit Work Plans accounting for any significant data gaps.

Action Items: Stone to review data gaps and revise Work Plan to account for all data gaps.

Consensus Decisions: Add metals analysis to certain GW and PW / SW samples, add soil samples to DPW SVI Investigation.

QAPP WORKSHEET #10: PROBLEM DEFINITION

☐ Worksheet Not Applicable (State Reason)

The problem to be addressed by the project: Address data gaps raised in the *Draft Data Gap Analysis Report*, dated May 25, 2012; Collect data necessary to support an RI, FS, Proposed Plan, and Decision Document, as outlined in the Performance Work Statement (PWS), issued by USACE on November 5, 2010 and subsequently modified on October 31 and December 12, 2011, and February 4, 2013.
The environmental questions being asked: What is the nature and extent of contamination? What are the risks from contamination? What is the best way to remediate contamination? Additional detail is provided in the Sections 3 and 4 of the Work Plan.
A synopsis of secondary data or information from site reports: Data are summarized in Section 2 of the Work Plan.
The possible classes of contaminants and the affected matrices: Chlorinated volatile organic compounds (CVOCs), petroleum VOCs, and metals in soils and groundwater, as well as surface water and possibly sediment.

QAPP WORKSHEET #11: PROJECT QUALITY OBJECTIVES/SYSTEMATIC PLANNING PROCESS STATEMENTS

☐ Worksheet Not Applicable (State Reason)

Who will use the data?
Data will be used by USACE, RIDEM, Stone Environmental, Inc., and Stone subcontractors. Data may also be used by USEPA and the Navy BRAC program.

What will the data be used for?
The data will be used to support the revised risk assessments for the *Final RI Report* and remedial decisions for the FS, *Proposed Plan*, and *Decision Document*. As part of the *Final RI Report*, the nature and extent of contamination will be further defined.

What types of data are needed? (target analytes, analytical groups, field screening, on-site analytical or off-site laboratory techniques, sampling techniques)
The investigation will include the following data:

- Groundwater: Volatile Organic Compounds (VOCs), total and dissolved Target Analyte List (TAL) metals, nitrate/nitrite, sulfate/sulfite, ferrous/ferric iron, chloride, ethane/ethene, Total Organic Carbon (TOC), TCE reductase (tceA), vinyl chloride reductase (bvcA), vinyl chloride reductase (vcrA), and *Dehalcoccoides* spp. (qDHC)
- Non-Aqueous Phase Liquid (NAPL) (oil samples): VOCs, SVOCs, Total Petroleum Hydrocarbons (TPH) Fuel Fingerprint, PCBs, Viscosity, and Specific Gravity
- Surface water: VOCs, Hardness, total and dissolved TAL metals
- Pore water: VOCs, total and dissolved TAL metals
- Rock core: Porosity
- Sediment: VOCs and TOC
- Soil Cores: NAPL detection, VOCs, TPH, fraction organic carbon (FOC).
- Residential Well Water: VOCs
- IDW (solid and liquid): VOCs, SVOCs, TPH, Pesticides, and PCBs

- Field screening will be conducted for the following groundwater analyses: nitrate/nitrite, sulfate/sulfite, ferrous/ferric iron, chloride, ethane/ethene.
- Mobile laboratory analysis will be conducted for VOA soil samples.
- Fixed laboratory analysis will be conducted for all other parameters.
- Standard operating procedures will be used for sample collection, handling, sample preparation, and analytical methods.
- All samples will be grab samples.
- All groundwater, surface water, and pore water will be sampled within as short a time frame as possible in order to provide synchronous results.

How "good" do the data need to be in order to support the environmental decision?
Fixed laboratory data needs to meet RIDEM and USACE FUDS requirements for data quality. The data need to be of sufficient quality to support the revised risk assessments for the *Final RI Report*, and remedial decisions for the FS, Proposed Plan, and Decision Document.

QAPP WORKSHEET #11: PROJECT QUALITY OBJECTIVES/SYSTEMATIC PLANNING PROCESS STATEMENTS (CONCLUDED)

☐ Worksheet Not Applicable (State Reason)

How many data are needed? (number of samples for each analytical group, matrix, and concentration)
Refer to Table 5 of SAP.
Groundwater samples:

- Existing Monitoring Wells 67 for VOCs, 32 for TAL metals (total and dissolved), 11 for qDHC, tceA, bvcA, and vcrA, 57 for nitrate/nitrite, sulfate/sulfite, ferrous/ferric iron, and chloride field screening, 65 for Ethane/ethane, 54 for TOC
- DPW Shallow Monitoring Wells 6 for VOCs.

Surface water samples: 17 for VOCs, 16 for Hardness, 10 for metals (total and dissolved)
Residential Well Water: 6 for VOCs
NAPL: Up to 4 for VOCs, SVOCs, TPH, PCBs, Fuel Finger Print, Viscosity, and Specific Gravity
Sediment: 17 for VOCs, 15 for TOC
Soil Cores: 585 for VOCs (Mobile laboratory) 10 for porosity & density, 11 for FOC, 14 for TPH
Pore water: 17 for VOCs, 10 for metals (total and dissolved)
Rock Core: 12 for Porosity
IDW: To be determined

Where, when, and how should the data be collected/generated?
Refer to Section 2 of SAP. Collection techniques include (numbers cross-reference to Worksheets #12-A through #12-Y):

1. Pore Water Sampling
2. Surface Water Sampling
3. Sediment Sampling
4. Soil Sampling
5. Groundwater Sampling
6. Drinking Water Sampling
7. Rock Core Sampling

Who will collect and generate the data?
Stone Environmental, Inc. and subcontracted laboratories.

How will the data be reported? The data will be included in the FS Report. The laboratories will submit analytical results in SEDD format, Level III data package deliverables with tabulated Form I results and spreadsheets (EDD).

How will the data be archived?
All original data will be electronically filed by Stone and retained for a period of ten years after the project has been closed. Final reports will be stored at a project repository.

QAPP WORKSHEET #12-A: MEASUREMENT PERFORMANCE CRITERIA

☐ Worksheet Not Applicable (State Reason)

Matrix	Soil Samples
Analytical Group	VOCs (SEI-Mobile)
Concentration Level	Low to High

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
21a	EPA Method 8260C/ASTMD6520, 27b	Precision	Relative percent difference (RPD) ≤ 30%	Laboratory Duplicates	A
		Precision	RPD ≤ 30%	Matrix Spike/Matrix Spike Duplicate (MS/MSD)	A
		Accuracy/Bias	Laboratory Control Limits[3]	Lab Control Sample (LCS)	A
		Precision-Overall/ Representativeness	RPD ≤ 50%	Field Duplicates	S&A
		Accuracy/Bias	No reported concentrations above ½ quantitation limits	Trip blank	S&A
		Accuracy/Bias	No reported concentrations above ½ quantitation limits	Equipment blank	S
		Accuracy/Bias	No reported concentrations above ½ quantitation limits	Method blank	A
		Accuracy/Bias	Laboratory control limits [3]	MS/MSD	A
		Accuracy	Laboratory control limits [3]	Surrogate: 4-Bromofluorobenzene	A

Note 1: See Worksheet #21 for cross-reference to sampling procedure.
Note 2: See Worksheet #23 for cross-reference to analytical method SOP.
Note 3: LCS and MS/MSD control limits may be compound-specific; individual control limits are provided in laboratory SOPs and for this project are the DOD limits.

QAPP WORKSHEET #12-B: MEASUREMENT PERFORMANCE CRITERIA

☐ Worksheet Not Applicable (State Reason)

Matrix	Aqueous Samples
Analytical Group	Natural Attenuation Parameters (SEI-Field)
Concentration Level	Low

[2]Hach Field Test Kit, Spectrophotometric
Nitrate – 8171; Nitrite – 8507; Sulfate – 8051; Sulfite – 8071; Ferrous Iron – 8146; Total Iron – 8008; Titration; Chloride – 8225. The methodologies for these procedures are found in the Hach *Water Chemistry Handbook, 5th Edition.*

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
6a, 15a	Hach Field Test Kit/12b	Precision	RPD ≤ 20%	Laboratory Duplicates	A
		Accuracy/Bias	+/-30% recovery[3]	Reference Sample (if applicable)	A
		Precision-Overall/ Representativeness	RPD ≤ 30%	Field Duplicates	S&A
		Accuracy/Bias	NA	Equipment blank	S
		Accuracy/Bias	No reported concentrations above ½ quantitation limits	Method blank	A

Note 1: See Worksheet #21 for cross-reference to sampling procedure.
Note 2: See Worksheet #23 for cross-reference to analytical method SOP.
Note 3: LCS control limits may be compound-specific; individual control limits are provided in laboratory SOPs.

QAPP WORKSHEET #12-C: MEASUREMENT PERFORMANCE CRITERIA

☐ Worksheet Not Applicable (State Reason)

Matrix	Aqueous Samples
Analytical Group	TCL VOCs (Fixed lab)
Concentration Level	Low

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
5a, 8a, 15a	EPA Method 8260B 4b, 5b	Precision	RPD ≤ 30%	Laboratory Duplicates	A
		Precision	RPD ≤ 30%	MS/MSD	A
		Accuracy/Bias	Laboratory control limits [3]	LCS	A
		Precision-Overall/ Representativeness	RPD ≤ 30%	Field Duplicates	S&A
		Accuracy/Bias	No reported concentrations above ½ quantitation limits	Trip blank/Equipment Blank	S&A
		Accuracy/Bias	No reported concentrations above ½ quantitation limits	Method blank	A
		Accuracy/Bias	Laboratory control limits [3]	MS/MSD	A
		Accuracy	70 to 120% recovery	Surrogate: 1,2-Dichloroethane-d4	A
		Accuracy	75 to 120% recovery	Surrogate: 4-Bromofluorobenzene	A
		Accuracy	85 to 115% recovery	Surrogate: Dibromofluoromethane	A
		Accuracy	85 to 120% recovery	Surrogate: Toluene-d8	A

Note 1: See Worksheet #21 for cross-reference to sampling procedure. Note 2: See Worksheet #23 for cross-reference to analytical method SOP.
Note 3: LCS and MS/MSD control limits may be compound-specific; individual control limits are provided in laboratory SOPs and are the DOD limits.

QAPP WORKSHEET #12-D: MEASUREMENT PERFORMANCE CRITERIA

☐ Worksheet Not Applicable (State Reason)

Matrix	Aqueous Samples
Analytical Group	Metals (Fixed lab)
Concentration Level	Low

Sampling Procedure[1]	**Analytical Method/SOP**[2]	**Data Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)**
5a, 8a, 15a	EPA Method 6010B/7470A/7000/ 6b, 7b, 8b, 9b	Precision	RPD ≤ 20%	Laboratory Duplicates	A
		Precision	RPD ≤ 20%	MS/MSD	A
		Accuracy/Bias	Laboratory control limits [3]	LCS	A
		Precision-Overall/ Representativeness	RPD ≤ 30%	Field Duplicates	S&A
		Accuracy/Bias	No reported concentrations above ½ quantitation limits	Equipment blank	S
		Accuracy/Bias	No reported concentrations above ½ quantitation limits	Method blank	A
		Accuracy/Bias	Laboratory control limits [3]	MS/MSD	A

Note 1: See Worksheet #21 for cross-reference to sampling procedure.
Note 2: See Worksheet #23 for cross-reference to analytical method SOP.
Note 3: LCS and MS/MSD control limits may be compound-specific; individual control limits are provided in laboratory SOPs (DOD limits).

QAPP WORKSHEET #12-E: MEASUREMENT PERFORMANCE CRITERIA

☐ Worksheet Not Applicable (State Reason)

Matrix	Aqueous Samples
Analytical Group	Ethane/ethene (Fixed lab)
Concentration Level	Low

Sampling Procedure[1]	**Analytical Method/SOP**[2]	**Data Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&**
5a, 8a, 15a	RSK 175/24b	Precision	RPD ≤ 30%	Laboratory Duplicates	A
		Precision	RPD ≤ 30%	MS/MSD	A
		Accuracy/Bias	Laboratory control limits [3]	LCS	A
		Precision-Overall/ Representativeness	RPD ≤ 30%	Field Duplicates	S&A
		Accuracy/Bias	No reported concentrations above ½ quantitation limits	Trip blank	S&A
		Accuracy/Bias	No reported concentrations above ½ quantitation limits	Equipment blank	S
		Accuracy/Bias	No reported concentrations above ½ quantitation limits	Method blank	A
		Accuracy/Bias	60 to 140% recovery[3]	MS/MSD	A

Note 1: See Worksheet #21 for cross-reference to sampling procedure.
Note 2: See Worksheet #23 for cross-reference to analytical method SOP.
Note 3: LCS and MS/MSD control limits may be compound-specific; individual control limits are provided in laboratory SOPs.

QAPP WORKSHEET #12-F: MEASUREMENT PERFORMANCE CRITERIA

☐ Worksheet Not Applicable (State Reason)

Matrix	Aqueous Samples
Analytical Group	TOC (Fixed lab)
Concentration Level	Low

Sampling Procedure[1]	**Analytical Method/SOP[2]**	**Data Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)**
5a, 8a, 15a	EPA Method 9060A/25b	Precision	RPD ≤ 20%	Laboratory Duplicates	A
		Precision	RPD ≤ 20%	MS/MSD	A
		Accuracy/Bias	+/-15% recovery	LCS	A
		Precision-Overall/ Representativeness	RPD ≤ 30%	Field Duplicates	S&A
		Accuracy/Bias	No reported concentrations above ½ quantitation limits	Equipment blank	S
		Accuracy/Bias	No reported concentrations above ½ quantitation limits	Method blank	A
		Accuracy/Bias	85 to 115% recovery	MS/MSD	A

Note 1: See Worksheet #21 for cross-reference to sampling procedure.
Note 2: See Worksheet #23 for cross-reference to analytical method SOP.

QAPP WORKSHEET #12-G: MEASUREMENT PERFORMANCE CRITERIA

☐ Worksheet Not Applicable (State Reason)

Matrix	reserved
Analytical Group	
Concentration Level	

Sampling Procedure[1]	**Analytical Method/SOP**[2]	**Data Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)**

Note 1: See Worksheet #21 for cross-reference to sampling procedure.
Note 2: See Worksheet #23 for cross-reference to analytical method SOP.

QAPP WORKSHEET #12-H: MEASUREMENT PERFORMANCE CRITERIA

☐ Worksheet Not Applicable (State Reason)

Matrix	reserved
Analytical Group	
Concentration Level	

Sampling Procedure[1]	**Analytical Method/SOP**[2]	**Data Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)**

Note 1: See Worksheet #21 for cross-reference to sampling procedure.
Note 2: See Worksheet #23 for cross-reference to analytical method SOP.

QAPP WORKSHEET #12-I: MEASUREMENT PERFORMANCE CRITERIA

☐ Worksheet Not Applicable (State Reason)

Matrix	Aqueous Samples
Analytical Group	qDHC, tceA, bvcA and vcrA (Fixed lab)
Concentration Level	Low

Dehalcoccoides spp. (qDHC) will be performed first. Should the preceding qDHC analyses indicate that microbial degradation is occurring, additional analyses for TCE reductase (tceA), vinyl chloride reductase (bvcA), vinyl chloride reductase (vcrA) will be performed by proprietary methods, in order to determine the specific biodegradation processes present

Sampling Procedure[1]	**Analytical Method/SOP[2]**	**Data Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)**
5a, 8a, 15a	Proprietary/28b	Precision	RPD ≤ 30%	Laboratory Duplicates	A
		Accuracy/Bias	TBD	LCS	A
		Precision-Overall/ Representativeness	RPD ≤ 30%	Field Duplicates	S&A
		Accuracy/Bias	TBD	Equipment blank	S
		Accuracy/Bias	No reported concentrations above ½ quantitation limits	Method blank	A

Note 1: See Worksheet #21 for cross-reference to sampling procedure.
Note 2: See Worksheet #23 for cross-reference to analytical method SOP.

QAPP WORKSHEET #12-J: MEASUREMENT PERFORMANCE CRITERIA

☐ Worksheet Not Applicable (State Reason)

Matrix	Aqueous Samples
Analytical Group	Hardness (Fixed lab)
Concentration Level	Low

Sampling Procedure[1]	**Analytical Method/SOP**[2]	**Data Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)**
5a, 8a, 15a	EPA Method 6010B/9b	Precision	RPD ≤ 20%	Laboratory Duplicates	A
		Precision	RPD ≤ 20%	MS/MSD	A
		Accuracy/Bias	Laboratory control limits [3]	LCS	A
		Precision-Overall/ Representativeness	RPD ≤ 30%	Field Duplicates	S&A
		Accuracy/Bias	No reported concentrations above ½ quantitation limits	Equipment blank	S
		Accuracy/Bias	No reported concentrations above ½ quantitation limits	Method blank	A
		Accuracy/Bias	80 to 120% recovery[3]	MS/MSD	A

Note 1: See Worksheet #21 for cross-reference to sampling procedure.
Note 2: See Worksheet #23 for cross-reference to analytical method SOP.
Note 3: LCS and MS/MSD control limits may be compound-specific; individual control limits are provided in laboratory SOPs.

QAPP WORKSHEET #12-K: MEASUREMENT PERFORMANCE CRITERIA

☐ Worksheet Not Applicable (State Reason)

Matrix	Aqueous (Drinking Water)
Analytical Group	VOCs (Fixed lab)
Concentration Level	Low

Sampling Procedure[1]	**Analytical Method/SOP**[2]	**Data Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)**
20a	EPA Method 524.2/13b	Precision	RPD ≤ 20%	Laboratory Duplicates	A
		Precision	RPD ≤ 20%	MS/MSD	A
		Accuracy/Bias	Laboratory control limits [3]	LCS	A
		Precision-Overall/ Representativeness	RPD ≤ 30%	Field Duplicates	S&A
		Accuracy/Bias	No reported concentrations above ½ quantitation limits	Trip blank	S&A
		Accuracy/Bias	No reported concentrations above ½ quantitation limits	Method blank	A
		Accuracy/Bias	70 to 130% recovery[3]	MS/MSD	A
		Accuracy	80 to 120% recovery	Surrogate: 1,2-Dichlorobenzene-d4	A
		Accuracy	80 to 120% recovery	Surrogate: 4-Bromofluorobenzene	A

Note 1: See Worksheet #21 for cross-reference to sampling procedure.
Note 2: See Worksheet #23 for cross-reference to analytical method SOP.
Note 3: LCS and MS/MSD control limits may be compound-specific; individual control limits are provided in laboratory SOPs.

QAPP WORKSHEET #12-L: MEASUREMENT PERFORMANCE CRITERIA

☐ Worksheet Not Applicable (State Reason)

Matrix	Sediment
Analytical Group	TCL VOCs (Fixed lab)
Concentration Level	Low

Sampling Procedure[1]	**Analytical Method/SOP[2]**	**Data Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)**
17a	EPA Method 8260B/ 4b, 5b	Precision	RPD ≤ 30%	Laboratory Duplicates	A
		Precision	RPD ≤ 30%	MS/MSD	A
		Accuracy/Bias	Laboratory control limits [3]	LCS	A
		Precision-Overall/ Representativeness	RPD ≤ 50%	Field Duplicates	S&A
		Accuracy/Bias	No reported concentrations above ½ quantitation limits	Trip blank/Equipment blank	S&A
		Accuracy/Bias	No reported concentrations above ½ quantitation limits	Method blank	A
		Accuracy/Bias	70 to 130% recovery[3]	MS/MSD	A
		Accuracy	70 to 120% recovery	Surrogate: 1,2-Dichloroethane-d4	A
		Accuracy	85 to 120% recovery	Surrogate: 4-Bromofluorobenzene	A
		Accuracy	85 to 115% recovery	Surrogate: Dibromofluoromethane	A
		Accuracy	85 to 115% recovery	Surrogate: Toluene-d8	A

Note 1: See Worksheet #21 for cross-reference to sampling procedure.
Note 2: See Worksheet #23 for cross-reference to analytical method SOP.
Note 3: LCS and MS/MSD control limits may be compound-specific; individual control limits are provided in laboratory SOPs.

QAPP WORKSHEET #12-M: MEASUREMENT PERFORMANCE CRITERIA

☐ Worksheet Not Applicable (State Reason)

Matrix	Sediment
Analytical Group	TOC (Fixed lab)
Concentration Level	Low

Sampling Procedure[1]	**Analytical Method/SOP**[2]	**Data Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)**
17a	Lloyd Kahn Method/25b	Precision	RPD ≤ 30%	Laboratory Duplicates	A
		Precision	RPD ≤ 30%	MS/MSD	A
		Accuracy/Bias	+/-30% recovery	LCS	A
		Precision-Overall/ Representativeness	RPD ≤ 50%	Field Duplicates	S&A
		Accuracy/Bias	No reported concentrations above ½ quantitation limits	Equipment blank	S
		Accuracy/Bias	No reported concentrations above ½ quantitation limits	Method blank	A
		Accuracy/Bias	70 to 130% recovery	MS/MSD	A

Note 1: See Worksheet #21 for cross-reference to sampling procedure.
Note 2: See Worksheet #23 for cross-reference to analytical method SOP.

QAPP WORKSHEET #12-N: MEASUREMENT PERFORMANCE CRITERIA

☐ Worksheet Not Applicable (State Reason)

Matrix	Soil
Analytical Group	Porosity and Density (Fixed lab)
Concentration Level	NA

Sampling Procedure[1]	**Analytical Method/SOP**[2]	**Data Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)**
17a, 21a	ASTM D7263 & ASTM D1298/ 1b, 2b	Precision	RPD ≤ 30%	Laboratory Duplicates	A
		Precision-Overall/ Representativeness	RPD ≤ 50%	Field Duplicates	S&A

Note 1: See Worksheet #21 for cross-reference to sampling procedure.
Note 2: See Worksheet #23 for cross-reference to analytical method SOP.

QAPP WORKSHEET #12-O: MEASUREMENT PERFORMANCE CRITERIA

☐ Worksheet Not Applicable (State Reason)

Matrix	Soil
Analytical Group	FOC (Fixed lab)
Concentration Level	Low

Sampling Procedure[1]	**Analytical Method/SOP**[2]	**Data Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)**
21a	ASTM D2974/3b	Precision	RPD ≤ 30%	Laboratory Duplicates	A
		Precision	RPD ≤ 30%	MS/MSD	A
		Accuracy/Bias	+/-30% recovery	LCS	A
		Precision-Overall/ Representativeness	RPD ≤ 50%	Field Duplicates	S&A
		Accuracy/Bias	No reported concentrations above ½ quantitation limits	Equipment blank	S
		Accuracy/Bias	No reported concentrations above quantitation limits	Method blank	A
		Accuracy/Bias	70 to 130% recovery	MS/MSD	A

Note 1: See Worksheet #21 for cross-reference to sampling procedure.
Note 2: See Worksheet #23 for cross-reference to analytical method SOP.

QAPP WORKSHEET #12-P: MEASUREMENT PERFORMANCE CRITERIA

☐ Worksheet Not Applicable (State Reason)

Matrix	Soil
Analytical Group	TPH (Fixed lab)
Concentration Level	Low

Sampling Procedure[1]	**Analytical Method/SOP**[2]	**Data Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)**
21a	8100M/22b	Precision	RPD ≤ 50%	Laboratory Duplicates	A
		Precision	RPD ≤ 50%	MS/MSD	A
		Accuracy/Bias	40 to 140% recovery[3]	LCS	A
		Precision-Overall/ Representativeness	RPD ≤ 50%	Field Duplicates	S&A
		Accuracy/Bias	No reported concentrations above ½ quantitation limits	Equipment blank	S
		Accuracy/Bias	No reported concentrations above quantitation limits	Method blank	A
		Accuracy/Bias	40 to 140% recovery[3]	MS/MSD	A
		Accuracy	40 to 140% recovery	Surrogate: O-Terphenyl	A

Note 1: See Worksheet #21 for cross-reference to sampling procedure.
Note 2: See Worksheet #23 for cross-reference to analytical method SOP.
Note 3: LCS and MS/MSD control limits may be compound-specific; individual control limits are provided in laboratory SOPs.

QAPP WORKSHEET #12-Q: MEASUREMENT PERFORMANCE CRITERIA

☐ Worksheet Not Applicable (State Reason)

Matrix	Rock
Analytical Group	Porosity (Fixed lab)
Concentration Level	na

Sampling Procedure[1]	**Analytical Method/SOP**[2]	**Data Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)**
21a	API RP-40/29b	Precision	RPD ≤ 30%	Laboratory Duplicates	A
		Precision-Overall/ Representativeness	RPD ≤ 50%	Field Duplicates	S&A

Note 1: See Worksheet #21 for cross-reference to sampling procedure.
Note 2: See Worksheet #23 for cross-reference to analytical method SOP.

QAPP WORKSHEET #12-R: MEASUREMENT PERFORMANCE CRITERIA

☐ Worksheet Not Applicable (State Reason)

Matrix	NAPL/IDW
Analytical Group	TCL VOCs (Fixed lab)
Concentration Level	High

Sampling Procedure[1]	**Analytical Method/SOP[2]**	**Data Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)**
23a	EPA Method 8260B 4b, 5b	Precision	RPD ≤ 30%	Laboratory Duplicates	A
		Precision	RPD ≤ 30%	MS/MSD	A
		Accuracy/Bias	Laboratory control limits [3]	LCS	A
		Precision-Overall/ Representativeness	RPD ≤ 50%	Field Duplicates	S&A
		Accuracy/Bias	No reported concentrations above ½ quantitation limits	Trip blank/Equipment blank	S&A
		Accuracy/Bias	No reported concentrations above ½ quantitation limits	Method blank	A
		Accuracy/Bias	70 to 130% recovery[3]	MS/MSD	A
		Accuracy	70 to 120% recovery	Surrogate: 1,2-Dichloroethane-d4	A
		Accuracy	85 to 120% recovery	Surrogate: 4-Bromofluorobenzene	A
		Accuracy	85 to 115% recovery	Surrogate: Dibromofluoromethane	A
		Accuracy	85 to 115% recovery	Surrogate: Toluene-d8	A

Note 1: See Worksheet #21 for cross-reference to sampling procedure.
Note 2: See Worksheet #23 for cross-reference to analytical method SOP.
Note 3: LCS and MS/MSD control limits may be compound-specific; individual control limits are provided in laboratory SOPs.

QAPP WORKSHEET #12-S: MEASUREMENT PERFORMANCE CRITERIA

☐ Worksheet Not Applicable (State Reason)

Matrix	NAPL/IDW
Analytical Group	SVOCs (Fixed lab)
Concentration Level	High

Sampling Procedure[1]	**Analytical Method/SOP[2]**	**Data Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)**
23a	EPA Method 8270C/23b	Precision	RPD ≤ 30%	Laboratory Duplicates	A
		Precision	RPD ≤ 30%	MS/MSD	A
		Accuracy/Bias	Laboratory control limits [3]	LCS	A
		Precision-Overall/ Representativeness	RPD ≤ 50%	Field Duplicates	S&A
		Accuracy/Bias	No reported concentrations above ½ quantitation limits	Equipment blank	S
		Accuracy/Bias	No reported concentrations above quantitation limits	Method blank	A
		Accuracy/Bias	40 to 140% recovery[3]	MS/MSD	A
		Accuracy	30 to 130% recovery	Surrogates: 1,2-Dichlorobenzene-d4, 2,4,6-Tribromophenol, 2-Chlorophenol-d4, 2-Fluorobiphenyl, 2-Fluorophenol, Nitrobenzene-d5, Phenol-d6, p-Terphenyl-d14	A

Note 1: See Worksheet #21 for cross-reference to sampling procedure. Note 2: See Worksheet #23 for cross-reference to analytical method SOP.
Note 3: LCS and MS/MSD control limits may be compound-specific; individual control limits are provided in laboratory SOPs (DOD Limits).

QAPP WORKSHEET #12-T: MEASUREMENT PERFORMANCE CRITERIA

☐ Worksheet Not Applicable (State Reason)

Matrix	NAPL/IDW
Analytical Group	Pesticides (Fixed lab)
Concentration Level	High

Sampling Procedure[1]	**Analytical Method/SOP**[2]	**Data Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)**
23a	EPA Method 8081A/ 20b	Precision	RPD ≤ 30%	Laboratory Duplicates	A
		Precision	RPD ≤ 30%	MS/MSD	A
		Accuracy/Bias	Laboratory control limits [3]	LCS	A
		Precision-Overall/ Representativeness	RPD ≤ 50%	Field Duplicates	S&A
		Accuracy/Bias	No reported concentrations above ½ quantitation limits	Equipment blank	S
		Accuracy/Bias	No reported concentrations above quantitation limits	Method blank	A
		Accuracy/Bias	70 to 125% recovery[3]	MS/MSD	A
		Accuracy	55 to 130% recovery	Surrogate: Decachlorobiphenyl	A
		Accuracy	70 to 125% recovery	Surrogate: Tetrachloro-m-xylene	A

Note 1: See Worksheet #21 for cross-reference to sampling procedure.
Note 2: See Worksheet #23 for cross-reference to analytical method SOP.
Note 3: LCS and MS/MSD control limits may be compound-specific; individual control limits are provided in laboratory SOPs (DOD Limits).

QAPP WORKSHEET #12-U: MEASUREMENT PERFORMANCE CRITERIA

☐ Worksheet Not Applicable (State Reason)

Matrix	NAPL/IDW
Analytical Group	PCBs (Fixed lab)
Concentration Level	High

Sampling Procedure[1]	**Analytical Method/SOP**[2]	**Data Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)**
23a	EPA Method 8082/ 21b	Precision	RPD ≤ 50%	Laboratory Duplicates	A
		Precision	RPD ≤ 50%	MS/MSD	A
		Accuracy/Bias	Laboratory control limits [3]	LCS	A
		Precision-Overall/ Representativeness	RPD ≤ 50%	Field Duplicates	S&A
		Accuracy/Bias	No reported concentrations above ½ quantitation limits	Equipment blank	S
		Accuracy/Bias	No reported concentrations above quantitation limits	Method blank	A
		Accuracy/Bias	40 to 140% recovery[3]	MS/MSD	A
		Accuracy	30 to 150% recovery	Surrogate: Decachlorobiphenyl	A
		Accuracy	30 to 150% recovery	Surrogate: Tetrachloro-m-xylene	A

Note 1: See Worksheet #21 for cross-reference to sampling procedure.
Note 2: See Worksheet #23 for cross-reference to analytical method SOP.
Note 3: LCS and MS/MSD control limits may be compound-specific; individual control limits are provided in laboratory SOPs (DOD Limits).

QAPP WORKSHEET #12-V: MEASUREMENT PERFORMANCE CRITERIA

☐ Worksheet Not Applicable (State Reason)

Matrix	NAPL/IDW
Analytical Group	Metals (Fixed lab)
Concentration Level	High

Sampling Procedure[1]	**Analytical Method/SOP**[2]	**Data Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)**
23a	EPA Method 6010B/7470A/7000/ 6b, 7b, 8b, 9b	Precision	RPD ≤ 20%	Laboratory Duplicates	A
		Precision	RPD ≤ 20%	MS/MSD	A
		Accuracy/Bias	Laboratory control limits [3]	LCS	A
		Precision-Overall/ Representativeness	RPD ≤ 50%	Field Duplicates	S&A
		Accuracy/Bias	No reported concentrations above ½ quantitation limits	Equipment blank	S
		Accuracy/Bias	No reported concentrations above quantitation limits	Method blank	A
		Accuracy/Bias	80 to 120% recovery[3]	MS/MSD	A

Note 1: See Worksheet #21 for cross-reference to sampling procedure.
Note 2: See Worksheet #23 for cross-reference to analytical method SOP.
Note 3: LCS and MS/MSD control limits may be compound-specific; individual control limits are provided in laboratory SOPs (DOD Limits).

QAPP WORKSHEET #12-W: MEASUREMENT PERFORMANCE CRITERIA

☐ Worksheet Not Applicable (State Reason)

Matrix	NAPL/IDW
Analytical Group	TPH & Petroleum Fingerprint (Fixed lab)
Concentration Level	High

Sampling Procedure[1]	**Analytical Method/SOP**[2]	**Data Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)**
23a	8100M/8015M/22b	Precision	RPD ≤ 50%	Laboratory Duplicates	A
		Precision	RPD ≤ 50%	MS/MSD	A
		Accuracy/Bias	40 to 140% recovery[3]	LCS	A
		Precision-Overall/ Representativeness	RPD ≤ 50%	Field Duplicates	S&A
		Accuracy/Bias	No reported concentrations above ½ quantitation limits	Equipment blank	S
		Accuracy/Bias	No reported concentrations above quantitation limits	Method blank	A
		Accuracy/Bias	40 to 140% recovery[3]	MS/MSD	A
		Accuracy	40 to 140% recovery	Surrogate: O-Terphenyl	A

Note 1: See Worksheet #21 for cross-reference to sampling procedure.
Note 2: See Worksheet #23 for cross-reference to analytical method SOP.
Note 3: LCS and MS/MSD control limits may be compound-specific; individual control limits are provided in laboratory SOPs.

QAPP WORKSHEET #12-X: MEASUREMENT PERFORMANCE CRITERIA

☐ Worksheet Not Applicable (State Reason)

Matrix	NAPL/IDW
Analytical Group	Viscosity (Fixed lab)
Concentration Level	High

Sampling Procedure[1]	**Analytical Method/SOP**[2]	**Data Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)**
23a	ASTM D 445-06 (@40°C)/26b	Precision	RPD ≤ 20%	Laboratory Duplicates	A
		Accuracy/Bias	+/-30% recovery[3]	Reference Sample (if applicable)	A
		Precision-Overall/ Representativeness	RPD ≤ 50%	Field Duplicates	S&A

Note 1: See Worksheet #21 for cross-reference to sampling procedure.
Note 2: See Worksheet #23 for cross-reference to analytical method SOP.

QAPP WORKSHEET #12-Y: MEASUREMENT PERFORMANCE CRITERIA

☐ Worksheet Not Applicable (State Reason)

Matrix	NAPL/IDW
Analytical Group	Specific Gravity (Fixed lab)
Concentration Level	High

Sampling Procedure[1]	**Analytical Method/SOP**[2]	**Data Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)**
23a	ASTM D287-92/14b	Precision	RPD ≤ 20%	Laboratory Duplicates	A
		Accuracy/Bias	+/-30% recovery[3]	Reference Sample (if applicable)	A
		Precision-Overall/ Representativeness	RPD ≤ 50%	Field Duplicates	S&A

Note 1: See Worksheet #21 for cross-reference to sampling procedure.
Note 2: See Worksheet #23 for cross-reference to analytical method SOP.

QAPP WORKSHEET #13: SECONDARY DATA CRITERIA AND LIMITATIONS TABLE

☐ Worksheet Not Applicable (State Reason)

Secondary Data	Data Source (Originating Organization, Report Title, and Date)	Data Generator(s) (Originating Org., Data Types, Data Generation/ Collection Dates)	How Data Will Be Used	Limitations on Data Use
Soil headspace screening	Stone, FS Report, 2013	Stone, October 2013	NAPL testing decisions	Qualitative
"Oil Red O" dye	Stone, FS Report, 2013	Stone, October 2013	NAPL presence decisions	Qualitative

QAPP WORKSHEET #14: SUMMARY OF PROJECT TASKS

☐ Worksheet Not Applicable (State Reason)

Sampling Tasks: Groundwater: Install additional wells, sample new and existing wells for analytical parameters listed in QAPP worksheet #11. Surface water: Sample twelve locations from the Hall Creek system and one in the northern wetlands for VOCs, Hardness and metals (total and dissolved). Pore water: Sample twelve locations from the Hall Creek system and one from northern wetlands for VOCs and metals (total and dissolved). Sediment: Sample twelve locations from the Hall Creek system and one in the northern wetlands for VOCs and TOC. Soil Cores: Sample soil cores from 30 locations in the Source Area for VOCs, TPH, and fraction of organic carbon (FOC). Rock Core: Sample four pre-existing rock cores for porosity analysis. NAPL: Verify presence of DNAPL in vicinity of Site monitoring well EA-102D, if present sample for analytical parameters listed in QAPP worksheet #11. Residential Well Water: Sample two residential, non-potable wells for VOCs.
Analysis Tasks: Stone Environmental, Inc. will analyze soil VOCs in a mobile laboratory, and perform field analyses of natural attenuation parameters. ESS Laboratory, et al., will perform fixed site lab analysis of all other samples.
Quality Control Tasks: Collect field duplicate samples, matrix spike and matrix-spike samples for certain aqueous, soil and rock samples.
Secondary Data: Secondary data will be used at a screening level only.
Data Management Tasks: Analytical data will be received from the laboratory using SEDD format and spreadsheets entered into the USACE ADR software for data review. Data will be uploaded to the EDMS database or other acceptable database in a format approved by USACE.
Documentation and Records: All new sampling locations will be marked for survey. Field forms will be used to record field sampling data, screening data and conditions at each sampling location. All chains of custody and shipping documents will be retained for the samples. Laboratory data packages will include a level 3 reporting package (results with QC/Calibrations). All documents will be filed in electronic format on Stone Environmental's secure local area network, which is backed up daily. Final documents will be stored at a Site specific repository.
Assessment/Audit Tasks: A field sample collection audit will be performed during the field work.
Data Review Tasks: Data and all related field logs/notes/records will be reviewed to assess total measurement error and determine overall usability of the data for project purposes. Data limitations will be determined and data will be compared to Project Quality Objectives and required method detection limits. Corrective action is initiated by the laboratory, as necessary. Final data are placed in database, with any necessary qualifiers, and tables, charts, and graphs are generated.

QAPP WORKSHEET #15-A: REFERENCE LIMITS AND EVALUATION TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix	Soil
Analytical Group	VOCs via EPA Method 8260C modified (SEI-Mobile)
Concentration Level	Low to High

Analyte	CAS Number	Project Action Limit (mg/kg)						Project Quantitation Limit (mg/kg)	Achievable Laboratory Detection Limits (mg/kg)	
		EPA Region III Residential RSL[1]	EPA Region III Industrial RSL[2]	RIDEM RSDEC[3]	RIDEM I/CSDEC[4]	RIDEM LCGA[5]	RIDEM LCGB[6]		MDL/LOD	RL/LOQ
Benzene	71-43-2	1.1	5.4	2.5	200	0.2	4.3	0.2	0.005	0.080
Chloroform	67-66-3	0.29	1.5	1.2	940	--	--	0.29	0.009	0.080
1,1-Dichloroethane	75-34-3	3.3	17	920	10,000	--	--	3.3	0.015	0.080
1,2-Dichloroethane	107-06-2	0.43	2.2	0.9	63	0.1	2.3	0.1	0.017	0.080
1,1-Dichloroethene	75-35-4	240	1,100	0.2	9.5	0.7	0.7	0.2	0.016	0.080
cis-1,2-Dichloroethene	156-59-2	160	2,000	630	10,000	1.7	60	1.7	0.008	0.080
trans-1,2-Dichloroethene	156-60-5	150	690	1,100	10,000	3.3	92	3.3	0.008	0.080
Ethylbenzene	100-41-4	5.4	27	71	10,000	27	62	5.4	0.024	0.080
Naphthalene	91-20-3	3.6	18	54	10,000	0.8	--	0.8	0.025	0.080
1,1,2,2-Tetrachloroethane	79-34-5	0.56	2.8	1.3	29	--	--	0.56	0.022	0.080
Tetrachloroethene	127-18-4	22	110	12	110	0.1	4.2	0.1	0.027	0.080
Toluene	108-88-3	5,000	45,000	190	10,000	32	54	32	0.007	0.080
1,1,2-Trichloroethane	79-00-5	1.1	5.3	3.6	100	0.1	--	0.1	0.020	0.080
Trichloroethene	79-01-6	0.91	6.4	13	560	0.2	20	0.2	0.008	0.080
1,2,4-Trimethylbenzene	95-63-6	62	260	--	--	--	--	62	0.023	0.080
1,3,5-Trimethylbenzene	108-67-8	780	10,000	--	--	--	--	780	0.022	0.080
Vinyl Chloride[9]	75-01-4	0.060	1.7	0.02	3.0	540	--	0.06	0.013	0.080
m- & p-Xylenes	1330-20-7	590[7]	2,500[7]	110[8]	10,000[8]	540	--	110	0.022[8]	0.080
o-Xylene	95-47-6	690	3,000	110[8]	10,000[8]	540	--	110	0.022	0.080

Note 1: USEPA Region III Regional Screening Levels for Residential Use.
Note 2: USEPA Region III Regional Screening Levels for Industrial Use.
Note 3: RIDEM Residential Soil Direct Exposure Criteria.
Note 4: RIDEM Industrial/Commercial Soil Direct Exposure Criteria.
Note 5: RIDEM Leachability Criteria GA Groundwater.
Note 6: RIDEM Leachability Criteria GB Groundwater.
Note 7: Lower of values for m- and p-Xylenes used.
Note 8: Values for total Xylenes used.
Note 9: Substance which has screening values below its LOQ and/or LOD. The selected analytical method is the best standard method available, and provides the lowest practical LOQ and LOD.

RIDEM standards are “To be Considered” values, and have been used to determine the Project Quantitation Limit only where they are above the Reporting Limit.

QAPP WORKSHEET #15-B: REFERENCE LIMITS AND EVALUATION TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix	Aqueous
Analytical Group	Natural Attenuation Parameters (SEI-Field)
Concentration Level	Low

Analyte	CAS Number	Project Action Limit (mg/L)			Project Quantitation Limit (mg/L)	Achievable Laboratory LOD (µg/L)
		Federal Maximum Contaminant Level	RIDEM Class GA[1]	RIDEM Class GB[2]		
Nitrate	14797-55-8	10,000	--	--	10	0.1
Nitrite	7632-00-0	1,000	--	--	1	0.002
Sulfate	14808-79-8	--	--	--	--	2.0
Sulfite	18785-72-3	--	--	--	--	0.005
Ferrous Iron	15438-31-0	--	--	--	--	0.02
Total Iron	7439-89-6	--	--	--	--	0.02
Chloride	16887-00-6	--	--	--	Not applicable	Not applicable

Note 1: RIDEM Numerical Groundwater Quality Standard, Class GA. Note 2: RIDEM Numerical Groundwater Quality Standard, Class GB.

RIDEM standards are “To be Considered” values, and have been used to determine the Project Quantitation Limit only where they are above the Reporting Limit.

QAPP WORKSHEET #15-C: REFERENCE LIMITS AND EVALUATION TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix	Aqueous
Analytical Group	TCL VOCs via EPA Method 8260B (Fixed Lab)
Concentration Level	Low

Analyte	CAS Number	Project Action Limits (µg/L)			Project Quantitation Limit (µg/L)	Achievable Laboratory Limits (µg/L)	
		EPA Region III Tap Water Regional Screening Levels[1]	RIDEM Groundwater Quality Standard Class GA[2]	EPA Region III Freshwater Screening Benchmark[3]		MDL/LOD	RL/LOQ
Acetone	67-64-1	12,000	--	1,500	1,500	2.7	10.0
Benzene[8]	71-43-2	0.39	5	370	0.39	0.1	1.0
Bromobenzene	108-86-1	54	--	--	54	0.2	2.0
Bromochloromethane	74-97-5	83	--	--	83	0.3	1.0
Bromodichloromethane[8]	75-27-4	0.12	--	--	0.12	0.1	0.6
Bromoform	75-25-2	7.9	--	320	7.9	0.2	1.0
Bromomethane	74-83-9	7.0	--	--	7.0	0.4	2.0
2-Butanone	78-93-3	4,900	--	14,000	4,900	3.4	10.0
n-Butylbenzene	104-51-8	780	--	--	780	0.1	1.0
sec-Butylbenzene	135-98-8	--	--	--	--	0.1	1.0
tert-Butylbenzene	98-06-6	--	--	--	--	0.1	1.0
Carbon Disulfide[8]	75-15-0	720	--	0.92	0.92	0.1	1.0
Carbon Tetrachloride[8]	56-23-5	0.39	5	13.3	0.39	0.1	1.0
Chlorobenzene	108-90-7	72	100	1.3	1.3	0.1	1.0
Chloroethane	75-00-3	--	--	--	--	0.4	2.0
Chloroform[8]	67-66-3	0.19	80[7]	1.8	0.19	0.2	1.0
1-Chlorohexane	544-10-5	--	--	--	--	0.4	1.0
Chloromethane	74-87-3	190	--	--	190	0.2	2.0
2-Chlorotoluene	95-49-8	--	--	--	--	0.1	1.0
4-Chlorotoluene	106-43-4	--	--	--	--	0.1	1.0
Dibromochloromethane[8]	124-48-1	0.15	--	--	0.15	0.2	1.0

Matrix	Aqueous
Analytical Group	TCL VOCs via EPA Method 8260B (Fixed Lab)
Concentration Level	Low

Analyte	CAS Number	Project Action Limits (µg/L)			Project Quantitation Limit (µg/L)	Achievable Laboratory Limits (µg/L)	
		EPA Region III Tap Water Regional Screening Levels[1]	RIDEM Groundwater Quality Standard Class GA[2]	EPA Region III Freshwater Screening Benchmark[3]		MDL/LOD	RL/LOQ
1,2-Dibromo-3-Chloropropane[8]	96-12-8	0.00032	0.2	--	0.00032	1.0	5.0
1,2-Dibromoethane[8]	106-93-4	0.0065	--	--	0.0065	0.2	1.0
Dibromomethane	74-95-3	7.9	--	--	7.9	0.3	1.0
1,2-Dichlorobenzene[8]	95-50-1	280	600	0.07	0.07	0.1	1.0
1,3-Dichlorobenzene	154-73-1	--	600	150	150	0.2	1.0
1,4-Dichlorobenzene[8]	106-46-7	0.42	75	26	0.42	0.1	1.0
1,1-Dichloroethane	75-34-3	2.4	--	47	2.4	0.2	1.0
1,2-Dichloroethane[8]	107-06-2	0.15	5	100	0.15	0.2	1.0
1,1-Dichloroethene	75-35-4	260	7	25	7	0.3	1.0
cis-1,2-Dichloroethene	156-59-2	28	70	2,400	28	0.2	1.0
trans-1,2-Dichloroethene	166-60-5	86	100	970	86	0.3	1.0
1,2-Dichloropropane[8]	78-87-5	0.38	5	3,000	0.38	0.2	1.0
1,3-Dichloropropane	142-28-9	290	--	--	290	0.1	1.0
2,2-Dichloropropane	594-20-7	--	--	--	--	0.3	1.0
1,1-Dichloropropene	563-58-6	--	--	--	--	0.2	2.0
cis-1,3-Dichloropropene[8]	10061-01-5	--	--	0.055[4]	0.055	0.2	0.4
trans-1,3-Dichloropropene[8]	10061-02-6	--	--	0.055[4]	0.055	0.2	0.4
Dichlorodifluoromethane	75-71-8	190	--	--	190	0.3	2.0
Diethyl Ether	60-29-7	--	--	--	--	0.3	1.0
Di-isopropyl ether	108-20-3	1,500	--	--	1,500	0.2	1.0
1,4-Dioxane - Screen[8]	123-91-1	0.67	--	--	0.67	190	500
Ethyl tertiary-butyl ether	637-92-3	--	--	--	--	0.1	1.0
Ethylbenzene	100-41-4	1.3	700	90	1.3	0.1	1.0
Hexachlorobutadiene[8]	87-68-3	0.26	--	1.3	0.26	0.2	0.6

Matrix	Aqueous
Analytical Group	TCL VOCs via EPA Method 8260B (Fixed Lab)
Concentration Level	Low

Analyte	CAS Number	Project Action Limits (µg/L)			Project Quantitation Limit (µg/L)	Achievable Laboratory Limits (µg/L)	
		EPA Region III Tap Water Regional Screening Levels[1]	RIDEM Groundwater Quality Standard Class GA[2]	EPA Region III Freshwater Screening Benchmark[3]		MDL/LOD	RL/LOQ
Hexachloroethane[8]	67-72-1	0.79	--	12	0.79	0.2	1.0
2-Hexanone	591-78-6	34	--	99	34	1.5	10.0
Isopropylbenzene	98-82-8	--	--	2.6	2.6	0.1	1.0
4-Isopropyltoluene	99-87-6	--	--	85	85	0.1	1.0
4-Methyl-2-Pentanone	108-10-1	--	--	170	170	1.6	10.0
Methyl tert-Butyl Ether	1634-04-4	12	40	11,070	12	0.3	1.0
Methylene Chloride	79-09-2	9.9	--	98.1	9.9	0.2	2.0
Naphthalene[8]	91-20-3	0.14	100	1.1	0.14	0.2	1.0
n-Propylbenzene	74296-31-4	530	--	128	128	0.2	1.0
Styrene	100-42-5	1,100	100	72	72	0.1	1.0
Tertiary-amyl methyl ether	994-05-8	--	--	--	--	0.2	1.0
1,1,1,2-Tetrachloroethane[8]	630-20-6	0.5	--	--	0.5	0.2	1.0
1,1,2,2-Tetrachloroethane[8]	79-34-5	0.066	--	610	0.066	0.1	0.5
Tetrachloroethene	127-18-4	9.7	5	610	9.7	0.2	1.0
Tetrahydrofuran	109-99-9	3,200	--	111	111	1.6	5.0
Toluene	108-88-3	860	1,000	2	2	0.1	1.0
1,2,3-Trichlorobenzene	87-61-6	5.2	--	8	5.2	0.2	1.0
1,2,4-Trichlorobenzene[8]	120-82-1	0.99	70	24	0.99	0.2	1.0
1,1,1-Trichloroethane	71-55-6	7,500	200	11	11	0.2	1.0
1,1,2-Trichloroethane[8]	79-00-5	0.24	5	1,200	0.24	0.2	1.0
Trichloroethene[8]	79-01-6	0.44	5	21	0.44	0.2	1.0
Trichlorofluoromethane	75-69-4	1,100	--	--	1,100	0.4	1.0
1,2,4-Trimethylbenzene	95-63-6	15	--	33	15	0.1	1.0
1,3,5-Trimethylbenzene	108-67-8	87	--	71	71	0.1	1.0
1,2,3-Trichloropropane[8]	96-18-4	0.00065	--	1,200	0.00065	0.3	1.0

Matrix	Aqueous
Analytical Group	TCL VOCs via EPA Method 8260B (Fixed Lab)
Concentration Level	Low

Analyte	CAS Number	Project Action Limits (µg/L)			Project Quantitation Limit (µg/L)	Achievable Laboratory Limits (µg/L)	
		EPA Region III Tap Water Regional Screening Levels[1]	RIDEM Groundwater Quality Standard Class GA[2]	EPA Region III Freshwater Screening Benchmark[3]		MDL/LOD	RL/LOQ
Vinyl Chloride[8]	75-01-4	0.015	2	930	0.015	0.2	1.0
o-Xylene	95-47-6	190	10,000[6]	13	13	0.1	1.0
m- & p-Xylenes	1330-20-7	190	10,000[6]	1.8[5], 13	1.8	0.2	2.0

Note 1: U.S. Environmental Protection Agency, Region III Regional Screening Level, dated May 2008, for tap water.
Note 2: RIDEM Numerical Groundwater Quality Standard, Class GA, for groundwater results.
Note 3: U.S. Environmental Protection Agency, Region III Biological Technical Assistance Group, Freshwater Screening Standards, dated July 2006, for pore water and surface water results.
Note 4: Value for total 1,3-dichloropropene used.
Note 5: Value for m-xylene is 1.8 µg/L; value for total xylenes is 13 µg/L.
Note 6: Value for total xylenes.
Note 7: Value for total trihalomethanes.
Note 8: Substance which has screening values below its LOQ and/or LOD. The selected analytical method is the best standard method available, and provides the lowest practical LOQ and LOD.

RIDEM standards are "To be Considered" values, and have been used to determine the Project Quantitation Limit only where they are above the Reporting Limit.

QAPP WORKSHEET #15-D: REFERENCE LIMITS AND EVALUATION TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix	Aqueous
Analytical Group	TAL Metals via EPA Method 6010B/7470A/7000 (Fixed Lab)
Concentration Level	Low

Analyte	CAS Number	Project Action Limit (µg/L)			Project Quantitation Limit (µg/L)	Achievable Laboratory Limits (µg/L)	
		EPA Region III Tap Water Regional Screening Levels	RIDEM GA[1]	RIDEM GB[2]		MDL/LOD	RL/LOQ
Aluminum	7429-90-5	16,000	--	--	16,000	13.0	50.0
Antimony	7440-36-0	6	6	--	6	0.6	2.5
Arsenic	7440-38-2	0.045	10	--	0.045	0.4	2.5
Barium	7440-39-3	2,900	2,000	--	2,000	1.5	25.0
Beryllium	7440-41-7	16	4	--	4	0.1	0.5
Cadmium	7440-43-9	6.9	5	--	5	0.5	2.5
Calcium	7440-70-2	--	--	--	--	32.5	100
Chromium	7440-47-3	--	100	--	100	1.5	10.0
Cobalt	7440-48-4.	4.7	--	--	4.7	1.0	10.0
Copper[3]	7440-50-8	620	1.3	--	1.3	2.0	10.0
Iron	7439-89-6	11,000	--	--	11,000	11.5	50.0
Lead	7439-92-1	--	15	--	15	2.5	10.0
Magnesium	7439-95-4	--	--	--	--	21.0	100.0
Manganese	7439-96-5	320	--	--	320	3.0	10.0
Mercury	7439-97-6	0.63	2	--	0.63	0.12	0.50
Nickel	7440-02-0	--	--	--	--	1.0	25.0
Potassium	7440-09-7	--	--	--	--	55.0	2500
Selenium	7782-49-2	78	50	--	50	8.5	25.0
Silver	7440-22-4	71	--	--	71	1.0	5.0
Sodium	7646-69-7	--	--	--	--	135.0	2500
Thallium[3]	7440-28-0	0.16	2	--	0.16	0.4	1.5
Vanadium	7440-62-2	78	--	--	78	1.0	10.0
Zinc	7440-66-6	4,700	--	--	4,700	4.5	10.0

Note 1: RIDEM Numerical Groundwater Quality Standard, Class GA Note 2: RIDEM Numerical Groundwater Quality Standard, Class GB.
Note 3: Substance which has screening values below its LOQ and/or LOD. The selected analytical method is the best standard method available, and provides the lowest practical LOQ and LOD.
RIDEM standards are "To be Considered" values, and have been used to determine the Project Quantitation Limit only where they are above the Reporting Limit.

QAPP WORKSHEET #15-E: REFERENCE LIMITS AND EVALUATION TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix	Aqueous
Analytical Group	Ethane/Ethene via RSK 175 (Fixed Lab)
Concentration Level	Low

Analyte	CAS Number	Project Action Limit (µg/L)			Project Quantitation Limit (µg/L)	Achievable Laboratory Limits (µg/L)	
		EPA Region III Tap Water Regional Screening Levels	RIDEM GA[1]	RIDEM GB[2]		MDL/LOD	RL/LOQ
Ethane	74-84-0	--	--	--	--	1.40	6.60
Ethene	9002-88-4	--	--	--	--	0.90	4.80

Note 1: RIDEM Numerical Groundwater Quality Standard, Class GA. Note 2: RIDEM Numerical Groundwater Quality Standard, Class GB.

RIDEM standards are "To be Considered" values, and have been used to determine the Project Quantitation Limit only where they are above the Reporting Limit.

QAPP WORKSHEET #15-F: REFERENCE LIMITS AND EVALUATION TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix	Aqueous
Analytical Group	TOC via EPA Method 9060A (Fixed Lab)
Concentration Level	Low

Analyte	CAS Number	Project Action Limit (mg/L)			Project Quantitation Limit (mg/L)	Achievable Laboratory Limits (mg/L)	
		EPA Region III Tap Water Regional Screening Levels	RIDEM GA[1]	RIDEM GB[2]		MDL/LOD	RL/LOQ
Total organic carbon	7440-44-0	--	--	--	--	0.100	1.00

Note 1: RIDEM Numerical Groundwater Quality Standard, Class GA. Note 2: RIDEM Numerical Groundwater Quality Standard, Class GB.

RIDEM standards are "To be Considered" values, and have been used to determine the Project Quantitation Limit only where they are above the Reporting Limit.

QAPP WORKSHEET #15-G: REFERENCE LIMITS AND EVALUATION TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix	reserved
Analytical Group	
Concentration Level	

Analyte	CAS Number	Project Action Limit (mg/L)			Project Quantitation Limit (mg/L)	Achievable Laboratory Limits (mg/L)	
		EPA Region III Tap Water Regional Screening Levels	RIDEM GA[1]	RIDEM GB[2]		MDL/LOD	RL/LOQ

Note 1: RIDEM Numerical Groundwater Quality Standard, Class GA. Note 2: RIDEM Numerical Groundwater Quality Standard, Class GB.

RIDEM standards are "To be Considered" values, and have been used to determine the Project Quantitation Limit only where they are above the Reporting Limit.

QAPP WORKSHEET #15-H: REFERENCE LIMITS AND EVALUATION TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix	reserved
Analytical Group	
Concentration Level	

Analyte	CAS Number	Project Action Limit (mg/L)			Project Quantitation Limit (mg/L)	Achievable Laboratory Limits (mg/L)	
		EPA Region III Tap Water Regional Screening Levels	RIDEM GA[1]	RIDEM GB[2]		MDL/LOD	RL/LOQ

Note 1: RIDEM Numerical Groundwater Quality Standard, Class GA. Note 2: RIDEM Numerical Groundwater Quality Standard, Class GB.

RIDEM standards are "To be Considered" values, and have been used to determine the Project Quantitation Limit only where they are above the Reporting Limit.

QAPP WORKSHEET #15-I: REFERENCE LIMITS AND EVALUATION TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix	Aqueous
Analytical Group	qDHC, tceA, bvcA and vcrA via proprietary methods (Fixed Lab)
Concentration Level	Low

Analyte	**CAS Number**	**Project Action Limit (µg/L)**			**Project Quantitation Limit (µg/L)**	**Achievable Laboratory Limits (µg/L)**	
		Federal Maximum Contaminant Limit	**RIDEM GA[1]**	**RIDEM GB[2]**		**MDL/LOD**	**RL/LOQ**
Dehalcoccoides spp.	NA	--	--	--	--	--	--
TCE reductase	NA	--	--	--	--	--	--
vinyl chloride reductase	NA	--	--	--	--	--	--
vinyl chloride reductase	NA	--	--	--	--	--	--

Note 1: RIDEM Numerical Groundwater Quality Standard, Class GA. Note 2: RIDEM Numerical Groundwater Quality Standard, Class GB.
NA = Not applicable.

These methods are intended to provide data to assess biological activity related to bioremediation.

RIDEM standards are "To be Considered" values, and have been used to determine the Project Quantitation Limit only where they are above the Reporting Limit.

QAPP WORKSHEET #15-J: REFERENCE LIMITS AND EVALUATION TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix	Aqueous
Analytical Group	Hardness via EPA Method 6010B (Fixed Lab)
Concentration Level	Low

Analyte	**CAS Number**	**Project Action Limit (µg/L)**			**Project Quantitation Limit (µg/L)**	**Achievable Laboratory Limits (µg/L)**	
		EPA Region III Tap Water Regional Screening Levels	**RIDEM GA[1]**	**RIDEM GB[2]**		**MDL/LOD**	**RL/LOQ**
Hardness	10312	--	--	--	--	100	1,000

Note 1: RIDEM Numerical Groundwater Quality Standard, Class GA. Note 2: RIDEM Numerical Groundwater Quality Standard, Class GB.

RIDEM standards are "To be Considered" values, and have been used to determine the Project Quantitation Limit only where they are above the Reporting Limit.

QAPP WORKSHEET #15-K: REFERENCE LIMITS AND EVALUATION TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix	Aqueous
Analytical Group	VOCs via EPA Method 524.2 (Fixed Lab)
Concentration Level	Low

Analyte	CAS Number	Project Action Limit (µg/L)			Project Quantitation Limit (µg/L)	Achievable Laboratory Limits (µg/L)	
		EPA Region III Tap Water Regional Screening Levels	RIDEM GA[1]	RIDEM GB[2]		MDL/LOD	RL/LOQ
Acetone	67-64-1	12,000	--	--	12,000	0.98	5.00
Benzene[5]	71-43-2	0.39	5	140	0.39	0.06	0.50
Bromobenzene	108-86-1	54	--	--	54	0.06	0.50
Bromochloromethane	74-97-5	83	--	--	83	0.11	0.50
Bromodichloromethane[5]	75-27-4	0.12	--	--	0.12	0.08	0.50
Bromoform	75-25-2	7.9	--	--	7.9	0.16	0.50
Bromomethane	74-83-9	7.0	--	--	7.0	0.11	0.50
2-Butanone	78-93-3	4,900	--	--	4,900	1.60	5.00
n-Butylbenzene	104-51-8	780	--	--	780	0.06	0.50
sec-Butylbenzene	135-98-8	--	--	--	--	0.08	0.50
tert-Butylbenzene	98-06-6	--	--	--	--	0.09	0.50
Carbon Disulfide	75-15-0	720	--	--	720	0.09	0.50
Carbon Tetrachloride[5]	56-23-5	0.39	5	70	0.39	0.09	0.50
Chlorobenzene	108-90-7	72	100	3,200	72	0.06	0.50
Chloroethane	75-00-3	--	--	--	--	0.12	0.50
Chloroform[5]	67-66-3	0.19	80[4]	--	0.19	0.08	0.50
Chloromethane	74-87-3	190	--	--	190	0.10	0.50
2-Chlorotoluene	95-49-8	--	--	--	--	0.10	0.50
4-Chlorotoluene	106-43-4	--	--	--	--	0.12	0.50
Dibromochloromethane[5]	124-48-1	0.15	--	--	0.15	0.10	0.40
1,2-Dibromo-3-Chloropropane[5]	96-12-8	0.00032	0.2	2	0.00032	0.66	2.00
1,2-Dibromoethane[5]	106-93-4	0.0065	--	--	0.0065	0.08	0.50
Dibromomethane	74-95-3	7.9	--	--	7.9	0.10	0.50
1,2-Dichlorobenzene	95-50-1	280	600		280	0.07	0.50

Matrix	Aqueous
Analytical Group	VOCs via EPA Method 524.2 (Fixed Lab)
Concentration Level	Low

Analyte	CAS Number	Project Action Limit (µg/L)			Project Quantitation Limit (µg/L)	Achievable Laboratory Limits (µg/L)	
		EPA Region III Tap Water Regional Screening Levels	RIDEM GA[1]	RIDEM GB[2]		MDL/LOD	RL/LOQ
1,3-Dichlorobenzene	154-73-1	--	600	--	600	0.06	0.50
1,4-Dichlorobenzene	106-46-7	0.42	75	--	0.42	0.08	0.50
Dichlorodifluoromethane	75-71-8	190	--	--	190	0.09	0.50
1,1-Dichloroethane	75-34-3	2.4	--	--	2.4	0.09	0.50
1,2-Dichloroethane[5]	107-06-2	0.15	5	110	0.15	0.13	0.50
1,1-Dichloroethene	75-35-4	260	7	7	7	0.15	0.50
cis-1,2-Dichloroethene	156-59-2	28	70	2,400	28	0.09	0.50
trans-1,2-Dichloroethene	156-60-5	86	100	2,800	86	0.07	0.50
1,2-Dichloropropane[5]	78-87-5	0.38	5	3,000	0.38	0.11	0.50
1,3-Dichloropropane	142-28-9	290	--	--	290	0.10	0.50
2,2-Dichloropropane	594-20-7	--	--	--	--	0.12	0.50
1,1-Dichloropropene	563-58-6	--	--	--	--	0.08	0.50
cis-1,3-Dichloropropene	10061-01-5	--	--	--	--	0.10	0.30
trans-1,3-Dichloropropene	10061-02-6	--	--	--	--	0.10	0.30
Diethyl Ether	60-29-7	--	--	--	--	0.17	1.0
Di-isopropyl ether	108-20-3	1,500	--	--	1,500	0.14	1.00
Ethyl tertiary-butyl ether	637-92-3	--	--	--	--	0.09	1.0
Ethylbenzene	100-41-4	1.3	700	1,600	1.3	0.07	0.50
Hexachlorobutadiene[5]	87-68-3	0.26	--	--	0.26	0.12	0.40
Hexachloroethane[5]	67-72-1	0.79	--	--	0.79	0.12	1.0
2-Hexanone	591-78-6	34	--	--	34	1.03	5.00
Isopropylbenzene	98-82-8	--	--	--	--	0.06	0.50
4-Isopropyltoluene	99-87-6	--	--	--	--	0.07	0.50
4-Methyl-2-Pentanone	108-10-1	--	--	--	--	0.98	5.00
Methyl tert-Butyl Ether	1634-04-4	12	40	5,000	12	0.10	0.50
Methylene Chloride	79-09-2	9.9	--	--	9.9	0.10	0.50
Naphthalene[5]	91-20-3	0.14	100	--	0.14	0.16	0.50
n-Propylbenzene	74296-31-4	530	--	--	530	0.10	0.50

Matrix	Aqueous
Analytical Group	VOCs via EPA Method 524.2 (Fixed Lab)
Concentration Level	Low

Analyte	CAS Number	Project Action Limit (µg/L)			Project Quantitation Limit (µg/L)	Achievable Laboratory Limits (µg/L)	
		EPA Region III Tap Water Regional Screening Levels	RIDEM GA[1]	RIDEM GB[2]		MDL/LOD	RL/LOQ
Styrene	100-42-5	1,100	100	2,200	100	0.06	0.50
Tertiary-amyl methyl ether	994-05-8	--	--	--	--	0.13	1.00
1,1,1,2-Tetrachloroethane	630-20-6	0.5	--	--	0.5	0.07	0.50
1,1,2,2-Tetrachloroethane[5]	79-34-5	0.066	--	--	0.066	0.11	0.40
Tetrachloroethene	127-18-4	9.7	5	150	5	0.18	0.50
Tetrahydrofuran	109-99-9	3,200	--	--	3,200	0.66	2.00
Toluene	108-88-3	860	1,000	1,700	1,000	0.06	0.50
1,2,3-Trichlorobenzene	87-61-6	5.2	--	--	5.2	0.08	0.50
1,2,4-Trichlorobenzene	120-82-1	0.99	--	--	0.99	0.07	0.50
1,1,1-Trichloroethane	71-55-6	7,500	200	3,100	200	0.09	0.50
1,1,2-Trichloroethane[5]	79-00-5	0.24	5	--	0.24	0.14	0.50
Trichloroethene[5]	79-01-6	0.44	5	540	0.44	0.10	0.50
Trichlorofluoromethane	75-69-4	1,100	--	--	1,100	0.09	0.50
1,2,4-Trimethylbenzene	95-63-6	15	--	--	15	0.07	0.50
1,3,5-Trimethylbenzene	108-67-8	87	--	--	87	0.07	0.50
1,2,3-Trichloropropane[5]	96-18-4	0.00065	--	--	0.00065	0.16	0.50
Vinyl Chloride[5]	75-01-4	0.015	2	--	0.015	0.06	0.20
o-Xylene	95-47-6	190	10,000[3]	--	190	0.07	0.50
m- & p-Xylenes	1330-20-7	190	10,000[3]	--	190	0.14	1.00

Note 1: RIDEM Numerical Groundwater Quality Standard, Class GA.
Note 2: RIDEM Numerical Groundwater Quality Standard, Class GB.
Note 3: Value for total xylenes.
Note 4: Value for total trihalomethanes.
Note 5: Substance which has screening values below its LOQ and/or LOD. The selected analytical method is the best standard method available, and provides the lowest practical LOQ and LOD.
RIDEM standards are "To be Considered" values, and have been used to determine the Project Quantitation Limit only where they are above the Reporting Limit.

QAPP WORKSHEET #15-L: REFERENCE LIMITS AND EVALUATION TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix	Soil (Sediment)
Analytical Group	VOCs via EPA Method 5035/8260B (Fixed Lab)
Concentration Level	Low

Analyte	CAS Number	Project Action Limit (mg/kg)					Project Quantitation Limit (mg/kg)	Achievable Laboratory Limits (mg/kg)	
		EPA Region III Residential RSL[1]	EPA Region III Industrial RSL[2]	RIDEM RSDEC[3]	RIDEM I/CSDEC[4]	USEPA Region III Freshwater Sediment Quality Standards		MDL/LOD	RL/LOQ
Acetone	67-64-1	61,000	630,000	7,800	10,000	--	7,800	0.0031	0.0050
Benzene	71-43-2	1.1	5.4	2.5	200	--	1.1	0.0017	0.0050
Bromobenzene	108-86-1	300	1,800	--	--	--	300	0.0016	0.0050
Bromochloromethane	74-97-5	160	680	--	--	--	160	0.0016	0.0050
Bromodichloromethane	75-27-4	0.27	1.4	10	92	--	0.27	0.0016	0.0050
Bromoform	75-25-2	62	220	81	720	0.654	0.654	0.0014	0.0050
Bromomethane	74-83-9	7.3	32	0.8	2,900	--	0.8	0.0017	0.0100
2-Butanone	78-93-3	28,000	200,000	--	--	--	28,000	0.0024	0.0050
n-Butylbenzene	104-51-8	3,900	51,000	--	--	--	3,900	0.0016	0.0050
sec-Butylbenzene	135-98-8	--	--	--	--	--	--	0.0016	0.0050
tert-Butylbenzene	98-06-6	--	--	--	--	--	--	0.0016	0.0050
Carbon Disulfide[9]	75-15-0	820	3,700	--	--	0.000851	0.000851	0.0016	0.0050
Carbon Tetrachloride	56-23-5	0.61	3.0	1.5	44	0.0642	0.0642	0.0016	0.0050
Chlorobenzene	108-90-7	290	1,400	210	10,000	0.00842	0.00842	0.0016	0.0050
Chloroethane	75-00-3	--	--	--	--	--	--	0.0018	0.0100
Chloroform	67-66-3	0.29	1.5	1.2	940	--	0.29	0.0017	0.0050
1-Chlorohexane	544-10-5	--	--	--	--	--	--	0.0016	0.0050
Chloromethane	74-87-3	120	500	--	--	--	120	0.0017	0.0100
2-Chlorotoluene	95-49-8	1,600	20,000	--	--	--	1,600	0.0010	0.0050
4-Chlorotoluene	106-43-4	1,600	20,000	--	--	--	1,600	0.0017	0.0050
Dibromochloromethane	124-48-1	0.68	3.3	7.6	68	--	0.68	0.0016	0.0050
1,2-Dibromo-3-Chloropropane	96-12-8	0.0054	0.069	0.5	4.1	--	0.01	0.0016	0.0050
1,2-Dibromoethane	106-93-4	0.034	0.17	--	--	--	0.03	0.0015	0.0050
Dibromomethane	74-95-3	25	110	--	--	--	25	0.0017	0.0050
1,2-Dichlorobenzene	95-50-1	1,900	9,800	510	10,000	0.0165	0.0165	0.0015	0.0050

Matrix	Soil (Sediment)
Analytical Group	VOCs via EPA Method 5035/8260B (Fixed Lab)
Concentration Level	Low

Analyte	CAS Number	Project Action Limit (mg/kg)					Project Quantitation Limit (mg/kg)	Achievable Laboratory Limits (mg/kg)	
		EPA Region III Residential RSL[1]	EPA Region III Industrial RSL[2]	RIDEM RSDEC[3]	RIDEM I/CSDEC[4]	USEPA Region III Freshwater Sediment Quality Standards		MDL/LOD	RL/LOQ
1,3-Dichlorobenzene	154-73-1	--	--	430	10,000	4.43	4.43	0.0016	0.0050
1,4-Dichlorobenzene	106-46-7	2.4	12	27	240	--	2.4	0.0015	0.0050
Dichlorodifluoromethane	75-71-8	94	400	--	--	--	94	0.0014	0.0100
1,1-Dichloroethane	75-34-3	3.3	17	920	10,000	--	3.3	0.0016	0.0050
1,2-Dichloroethane	107-06-2	0.43	2.2	0.9	63	--	0.43	0.0016	0.0050
1,1-Dichloroethene	75-35-4	240	1,100	0.2	9.5	0.031	0.031	0.0016	0.0050
cis-1,2-Dichloroethene	156-59-2	160	2,000	630	10,000		160	0.0017	0.0050
trans-1,2-Dichloroethene	156-60-5	150	690	1,100	10,000	1.05	1.05	0.0017	0.0050
1,2-Dichloropropane	78-87-5	0.94	4.7	1.9	84	--	0.94	0.0017	0.0050
1,3-Dichloropropane	142-28-9	1,600	20,000	--	--	--	1,600	0.0015	0.0050
2,2-Dichloropropane	594-20-7	--	--	--	--	--	--	0.0016	0.0050
1,1-Dichloropropene	563-58-6	--	--	--	--	--	--	0.0016	0.0050
cis-1,3-Dichloropropene[9]	10061-01-5	1.7[5]	8.3[5]	--	--	0.0000509[5]	0.0000509	0.0017	0.0050
trans-1,3-Dichloropropene[9]	10061-02-6	1.7[5]	8.3[5]	--	--	0.0000509[5]	0.0000509	0.0016	0.0050
Ethylbenzene	100-41-4	5.4	27	71	10,000	1.1	1.1	0.0016	0.0050
Hexachlorobutadiene	87-68-3	6.2	22	8.2	73	--	6.2	0.0015	0.0050
2-Hexanone	591-78-6	210	1,400	--	--	--	210	0.0036	0.0050
Isopropylbenzene	98-82-8	--	--	27	10,000	0.086	0.086	0.0017	0.0050
4-Isopropyltoluene	99-87-6	--	--	--	--	--	--	0.0016	0.0050
4-Methyl-2-Pentanone	108-10-1	5,300	53,000	--	--	--	5,300	0.0013	0.0500
Methyl tert-Butyl Ether	1634-04-4	43	220	390	10,000	--	43	0.0017	0.0050
Methylene Chloride	79-09-2	56	960	45	760	--	45	0.0019	0.0250
Naphthalene	91-20-3	3.6	18	54	10,000	0.176	0.176	0.0013	0.0050
n-Propylbenzene	74296-31-4	3,400	21,000			--	3,400	0.0012	0.0050

Matrix	Soil (Sediment)
Analytical Group	VOCs via EPA Method 5035/8260B (Fixed Lab)
Concentration Level	Low

Analyte	CAS Number	Project Action Limit (mg/kg)					Project Quantitation Limit (mg/kg)	Achievable Laboratory Limits (mg/kg)	
		EPA Region III Residential RSL[1]	EPA Region III Industrial RSL[2]	RIDEM RSDEC[3]	RIDEM I/CSDEC[4]	USEPA Region III Freshwater Sediment Quality Standards		MDL/LOD	RL/LOQ
Styrene	100-42-5	6,300	36,000	13	190	0.559	0.559	0.0015	0.0050
1,1,1,2-Tetrachloroethane	630-20-6	1.9	9.3	2.2	220	--	1.9	0.0015	0.0050
1,1,2,2-Tetrachloroethane	79-34-5	0.56	2.8	1.3	29	1.36	0.56	0.0015	0.0050
Tetrachloroethene	127-18-4	22	110	12	110	0.468	0.468	0.0017	0.0050
Toluene	108-88-3	5,000	45,000	190	10,000	2	2	0.0016	0.0050
1,2,3-Trichlorobenzene	87-61-6	49	490	--	--	0.858	0.858	0.0014	0.0050
1,2,4-Trichlorobenzene	120-82-1	22	99	96	10,000	2.1	2.1	0.0013	0.0050
1,1,1-Trichloroethane	71-55-6	8,700	38,000	--	--	0.0302	0.0302	0.0016	0.0050
1,1,2-Trichloroethane	79-00-5	1.1	5.3	3.6	100	1.24	1.1	0.0017	0.0050
Trichloroethene	79-01-6	0.91	6.4	13	560	0.0969	0.91	0.0017	0.0050
Trichlorofluoromethane	75-69-4	790	3,400	--	--	--	790	0.0017	0.0050
1,2,4-Trimethylbenzene	95-63-6	62	260	--	--	--	62	0.0016	0.0050
1,3,5-Trimethylbenzene	108-67-8	780	10,000	--	--	--	780	0.0016	0.0050
Vinyl Chloride	75-01-4	0.06	1.7	0.02	3.0	--	0.02	0.0017	0.0100
m- & p-Xylenes	1330-20-7	590[5]	2,500[5]	110[6]	10,000[6]	0.0252[6]	0.0252	0.0032	0.0100
o-Xylene[5]	95-47-6	690	3,000	110[6]	10,000[6]	0.0252[6]	0.0252	0.0016	0.0050

Note 1: USEPA Region III Regional Screening Levels for Residential Use.
Note 2: USEPA Region III Regional Screening Levels for Industrial Use.
Note 3: RIDEM Residential Soil Direct Exposure Criteria.
Note 4: RIDEM Industrial/Commercial Soil Direct Exposure Criteria.
Note 5: Value for total 1,2-Dichloropropane used.
Note 6: Lower of values for m- and p-Xylenes used.
Note 7: Value for total xylenes.
Note 8: Value for m-Xylene.
Note 9: Substance which has screening values below its LOQ and/or LOD. The selected analytical method is the best standard method available, and provides the lowest practical LOQ and LOD.
RIDEM standards are "To be Considered" values, and have been used to determine the Project Quantitation Limit only where they are above the Reporting Limit.

QAPP WORKSHEET #15-M: REFERENCE LIMITS AND EVALUATION TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix	Soil (Sediment)
Analytical Group	TOC via Lloyd Kahn Method (Fixed Lab)
Concentration Level	Low

Analyte	**CAS Number**	**Project Action Limit (mg/kg)**			**Project Quantitation Limit (mg/kg)**	**Achievable Laboratory Limits (mg/kg)**	
		EPA Region III Residential RSL[1]	**RIDEM RSDEC[2]**	**USEPA Region 3 Freshwater Sediment Quality Standards**		**MDL/LOD**	**RL/LOQ**
Total organic carbon	7440-44-0	--	--	--	--	1,000	--

Note 1: USEPA Region III Regional Screening Levels for Residential Use.

Note 2: RIDEM Residential Soil Direct Exposure Criteria.

RIDEM standards are "To be Considered" values, and have been used to determine the Project Quantitation Limit only where they are above the Reporting Limit.

QAPP WORKSHEET #15-N: REFERENCE LIMITS AND EVALUATION TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix	Soil
Analytical Group	Porosity and Density via ASTM D7263 & ASTM D1298/ (Fixed Lab)
Concentration Level	na

Analyte	CAS Number	Project Action Limit		Project Quantitation Limit	Achievable Laboratory LOD
		EPA Region III Residential RSL[1]	RIDEM RSDEC[2]		
Porosity	NA	--	--	--	NA
Density	NA	--	--	--	0.1 lb/ft^3

Note 1: USEPA Region III Regional Screening Levels for Residential Use.

Note 2: RIDEM Residential Soil Direct Exposure Criteria.

NA = Not applicable.

RIDEM standards are "To be Considered" values, and have been used to determine the Project Quantitation Limit only where they are above the Reporting Limit.

QAPP WORKSHEET #15-O: REFERENCE LIMITS AND EVALUATION TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix	Soil
Analytical Group	FOC via ASTM D2974 (Fixed Lab)
Concentration Level	na

Analyte	CAS Number	Project Action Limit		Project Quantitation Limit	Achievable Laboratory LOD
		EPA Region III Residential RSL[1]	RIDEM RSDEC[2]		
Fraction organic carbon	NA	--	--	--	0.1%

Note 1: USEPA Region III Regional Screening Levels for Residential Use.

Note 2: RIDEM Residential Soil Direct Exposure Criteria.

FOC: Fraction of Organic Carbon.

NA = Not applicable.

RIDEM standards are "To be Considered" values, and have been used to determine the Project Quantitation Limit only where they are above the Reporting Limit.

QAPP WORKSHEET #15-P: REFERENCE LIMITS AND EVALUATION TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix	Soil
Analytical Group	TPH via EPA Method 8100M (Fixed Lab)
Concentration Level	na

Analyte	CAS Number	Project Action Limit (mg/kg)		Project Quantitation Limit (mg/kg)	Achievable Laboratory Limits (mg/kg)	
		EPA Region III Residential RSL[1]	RIDEM RSDEC[2]		MDL/LOD	RL/LOQ
TPH	NA	--	500	500	5.6	37.5

Note 1: USEPA Region III Regional Screening Levels for Residential Use.

Note 2: RIDEM Residential Soil Direct Exposure Criteria.

NA = Not applicable.

RIDEM standards are “To be Considered” values, and have been used to determine the Project Quantitation Limit only where they are above the Reporting Limit.

QAPP WORKSHEET #15-Q: REFERENCE LIMITS AND EVALUATION TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix	Rock
Analytical Group	Porosity via API RP-40 (Fixed Lab)
Concentration Level	na

Analyte	CAS Number	Project Action Limit		Project Quantitation Limit	Achievable Laboratory LOD
		EPA Region III Residential RSL[1]	RIDEM RSDEC[2]		
Porosity	NA	--	--	0.5%	0.5%

Note 1: USEPA Region III Regional Screening Levels for Residential Use.

Note 2: RIDEM Residential Soil Direct Exposure Criteria.

NA = Not applicable.

RIDEM standards are "To be Considered" values, and have been used to determine the Project Quantitation Limit only where they are above the Reporting Limit.

QAPP WORKSHEET #15-R: REFERENCE LIMITS AND EVALUATION TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix	NAPL/IDW
Analytical Group	VOCs via EPA Method 5035/8260B (Fixed Lab)
Concentration Level	High

Analyte	CAS Number	Project Action Limit (mg/kg)			Project Quantitation Limit (mg/kg)	Achievable Laboratory Limits (mg/kg)	
		None	None	None		MDL/LOD	RL/LOQ
Acetone	67-64-1	--	--	--	--	370	1250
Benzene	71-43-2	--	--	--	--	8.1	50.0
Bromobenzene	108-86-1	--	--	--	--	13.7	50.0
Bromochloromethane	74-97-5	--	--	--	--	16.2	50.0
Bromodichloromethane	75-27-4	--	--	--	--	6.9	50.0
Bromoform	75-25-2	--	--	--	--	14.4	50.0
Bromomethane	74-83-9	--	--	--	--	33.4	100
2-Butanone	78-93-3	--	--	--	--	289	1250
n-Butylbenzene	104-51-8	--	--	--	--	12.3	50.0
sec-Butylbenzene	135-98-8	--	--	--	--	6.7	50.0
tert-Butylbenzene	98-06-6	--	--	--	--	11.7	50.0
Carbon Disulfide	75-15-0	--	--	--	--	7.4	50.0
Carbon Tetrachloride	56-23-5	--	--	--	--	8.7	50.0
Chlorobenzene	108-90-7	--	--	--	--	7.9	50.0
Chloroethane	75-00-3	--	--	--	--	33.3	100
Chloroform	67-66-3	--	--	--	--	10.3	50.0
Chloromethane	74-87-3	--	--	--	--	12.7	100
2-Chlorotoluene	95-49-8	--	--	--	--	14.1	50.0
4-Chlorotoluene	106-43-4	--	--	--	--	6.5	50.0
Dibromochloromethane	124-48-1	--	--	--	--	12.6	50.0
1,2-Dibromo-3-Chloropropane	96-12-8	--	--	--	--	100	300
1,2-Dibromoethane	106-93-4	--	--	--	--	12.7	50.0
Dibromomethane	74-95-3	--	--	--	--	15.8	50.0
1,2-Dichlorobenzene	95-50-1	--	--	--	--	7.1	50.0
1,3-Dichlorobenzene	154-73-1	--	--	--	--	6.3	50.0
1,4-Dichlorobenzene	106-46-7	--	--	--	--	13.3	50.0

Matrix	NAPL/IDW
Analytical Group	VOCs via EPA Method 5035/8260B (Fixed Lab)
Concentration Level	High

Analyte	CAS Number	Project Action Limit (mg/kg)			Project Quantitation Limit (mg/kg)	Achievable Laboratory Limits (mg/kg)	
		None	None	None		MDL/LOD	RL/LOQ
Dichlorodifluoromethane	75-71-8	--	--	--	--	8.7	50.0
1,1-Dichloroethane	75-34-3	--	--	--	--	8.0	50.0
1,2-Dichloroethane	107-06-2	--	--	--	--	13.4	50.0
1,1-Dichloroethene	75-35-4	--	--	--	--	12.3	50.0
cis-1,2-Dichloroethene	156-59-2	--	--	--	--	12.4	50.0
trans-1,2-Dichloroethene	156-60-5	--	--	--	--	16.4	50.0
1,2-Dichloropropane	78-87-5	--	--	--	--	13.1	50.0
1,3-Dichloropropane	142-28-9	--	--	--	--	11.2	50.0
2,2-Dichloropropane	594-20-7	--	--	--	--	17.1	100
1,1-Dichloropropene	563-58-6	--	--	--	--	7.7	50.0
cis-1,3-Dichloropropene	10061-01-5	--	--	--	--	11.3	50.0
trans-1,3-Dichloropropene	10061-02-6	--	--	--	--	15.4	50.0
Ethylbenzene	100-41-4	--	--	--	--	6.5	50.0
Hexachlorobutadiene	87-68-3	--	--	--	--	16.7	50.0
2-Hexanone	591-78-6	--	--	--	--	86.1	500
Isopropylbenzene	98-82-8	--	--	--	--	8.8	50.0
4-Isopropyltoluene	99-87-6	--	--	--	--	8.9	50.0
4-Methyl-2-Pentanone	108-10-1	--	--	--	--	60.2	500
Methyl tert-Butyl Ether	1634-04-4	--	--	--	--	8.0	50.0
Methylene Chloride	79-09-2	--	--	--	--	13.1	250
Naphthalene	91-20-3	--	--	--	--	13.1	50.0
n-Propylbenzene	74296-31-4	--	--	--	--	12.2	50.0
Styrene	100-42-5	--	--	--	--	6.6	50.0
1,1,1,2-Tetrachloroethane	630-20-6	--	--	--	--	8.7	100
1,1,2,2-Tetrachloroethane	79-34-5	--	--	--	--	13.6	50.0
Tetrachloroethene	127-18-4	--	--	--	--	16.7	50.0
Toluene	108-88-3	--	--	--	--	12.7	50.0
1,2,3-Trichlorobenzene	87-61-6	--	--	--	--	16.7	50.0
1,2,4-Trichlorobenzene	120-82-1	--	--	--	--	11.0	50.0

Matrix	NAPL/IDW
Analytical Group	VOCs via EPA Method 5035/8260B (Fixed Lab)
Concentration Level	High

Analyte	CAS Number	Project Action Limit (mg/kg)			Project Quantitation Limit (mg/kg)	Achievable Laboratory Limits (mg/kg)	
		None	None	None		MDL/LOD	RL/LOQ
1,1,1-Trichloroethane	71-55-6	--	--	--	--	8.8	50.0
1,1,2-Trichloroethane	79-00-5	--	--	--	--	12.5	50.0
Trichloroethene	79-01-6	--	--	--	--	10.3	50.0
Trichlorofluoromethane	75-69-4	--	--	--	--	13.2	50.0
1,2,3-Trichloropropane	96-18-4	--	--	--	--	12.4	50.0
1,2,4-Trimethylbenzene	95-63-6	--	--	--	--	9.6	50.0
1,3,5-Trimethylbenzene	108-67-8	--	--	--	--	8.8	50.0
Vinyl Chloride	75-01-4	--	--	--	--	16.5	50.0
m- & p-Xylenes[3]	1330-20-7	--	--	--	--	19.4	100
o-Xylene	95-47-6	--	--	--	--	9.6	50.0

PALs are not applicable to free product (NAPL) or Investigation Derived Waste.

QAPP WORKSHEET #15-S: REFERENCE LIMITS AND EVALUATION TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix	NAPL/IDW
Analytical Group	SVOCs via EPA Method 8270 (Fixed Lab)
Concentration Level	High

Analyte	CAS Number	Project Action Limit (mg/kg)			Project Quantitation Limit (mg/kg)	Achievable Laboratory Limits (mg/kg)	
		None	None	None		MDL/LOD	RL/LOQ
1,1-Biphenyl	92-52-4	--	--	--	--	24	100
1,2,4,5-Tetrachlorobenzene	95-94-3	--	--	--	--	23	100
1,2,4-Trichlorobenzene	120-82-1	--	--	--	--	27	100
1,2-Dichlorobenzene	95-50-1	--	--	--	--	26	100
1,3-Dichlorobenzene	541-73-1	--	--	--	--	24	100
1,4-Dichlorobenzene	106-46-7	--	--	--	--	25	100
2,3,4,6-Tetrachlorophenol	58-90-2	--	--	--	--	30	501
2,4,5-Trichlorophenol	95-95-4	--	--	--	--	20	100
2,4,6-Trichlorophenol	88-06-2	--	--	--	--	25	100
2,4-Dichlorophenol	120-83-2	--	--	--	--	25	100
2,4-Dimethylphenol	105-67-9	--	--	--	--	23	100
2,4-Dinitrophenol	51-28-5	--	--	--	--	167	501
2,4-Dinitrotoluene	121-14-2	--	--	--	--	32	100
2,6-Dinitrotoluene	606-20-2	--	--	--	--	24	100
2-Chloronaphthalene	91-58-7	--	--	--	--	22	100
2-Chlorophenol	95-57-8	--	--	--	--	28	100
2-Methylnaphthalene	91-57-6	--	--	--	--	22	100
2-Methylphenol	95-48-7	--	--	--	--	25	100
2-Nitroaniline	88-74-4	--	--	--	--	21	100
2-Nitrophenol	88-75-5	--	--	--	--	25	100
3,3´-Dichlorobenzidine	91-94-1	--	--	--	--	22	200
3+4-Methylphenol	106-44-5	--	--	--	--	53	200
3-Nitroaniline	99-09-2	--	--	--	--	26	100
4,6-Dinitro-2-Methylphenol	534-52-1	--	--	--	--	98	501
4-Bromophenyl-phenylether	101-55-3	--	--	--	--	19	100
4-Chloro-3-Methylphenol	59-50-7	--	--	--	--	29	100
4-Chloroaniline	106-47-8	--	--	--	--	23	200

Matrix	NAPL/IDW
Analytical Group	SVOCs via EPA Method 8270 (Fixed Lab)
Concentration Level	High

Analyte	CAS Number	Project Action Limit (mg/kg)			Project Quantitation Limit (mg/kg)	Achievable Laboratory Limits (mg/kg)	
		None	None	None		MDL/LOD	RL/LOQ
4-Chloro-phenyl-phenyl ether	7005-72-3	--	--	--	--	32	100
4-Nitroaniline	100-01-6	--	--	--	--	22	100
4-Nitrophenol	100-02-7	--	--	--	--	143	501
Acenaphthene	83-32-9	--	--	--	--	24	100
Acenaphthylene	208-96-8	--	--	--	--	19	100
Acetophenone	98-86-2	--	--	--	--	25	200
Aniline	62-53-3	--	--	--	--	25	501
Anthracene	120-12-7	--	--	--	--	16	100
Azobenzene	103-33-3	--	--	--	--	20	100
Benzidine	119-90-4	--	--	--	--	67	200
Benzo(a)anthracene	56-55-3	--	--	--	--	17	100
Benzo(a)pyrene	50-32-8	--	--	--	--	17	50
Benzo(b)fluoranthene	205-99-2	--	--	--	--	22	100
Benzo(g,h,i)perylene	191-24-2	--	--	--	--	22	100
Benzo(k)fluoranthene	207-08-9	--	--	--	--	29	100
Benzoic Acid	65-85-0	--	--	--	--	103	501
Benzyl Alcohol	100-51-6	--	--	--	--	33	100
bis(2-Chloroethoxy)methane	111-91-1	--	--	--	--	23	100
bis(2-Chloroethyl)ether	111-44-4	--	--	--	--	27	100
bis(2-chloroisopropyl)Ether	108-60-1	--	--	--	--	27	100
bis(2-Ethylhexyl)phthalate	117-81-7	--	--	--	--	33	100
Butylbenzylphthalate	85-68-7	--	--	--	--	20	100
Carbazole	86-74-8	--	--	--	--	17	100
Chrysene	218-01-9	--	--	--	--	17	50
Dibenzo(a,h)Anthracene	53-70-3	--	--	--	--	15	50
Dibenzofuran	132-64-9	--	--	--	--	20	100
Diethylphthalate	84-66-2	--	--	--	--	27	100
Dimethylphthalate	131-11-3	--	--	--	--	23	100
Di-n-butylphthalate	84-74-2	--	--	--	--	16	100

Matrix	NAPL/IDW
Analytical Group	SVOCs via EPA Method 8270 (Fixed Lab)
Concentration Level	High

Analyte	CAS Number	Project Action Limit (mg/kg)			Project Quantitation Limit (mg/kg)	Achievable Laboratory Limits (mg/kg)	
		None	None	None		MDL/LOD	RL/LOQ
Di-n-octylphthalate	117-84-0	--	--	--	--	17	100
Fluoranthene	206-44-0	--	--	--	--	17	100
Fluorene	86-73-7	--	--	--	--	26	100
Hexachlorobenzene	118-74-1	--	--	--	--	17	100
Hexachlorobutadiene	87-68-3	--	--	--	--	26	100
Hexachlorocyclopentadiene	77-47-4	--	--	--	--	115	501
Hexachloroethane	67-72-1	--	--	--	--	25	100
Indeno(1,2,3-cd)Pyrene	193-39-5	--	--	--	--	32	100
Isophorone	78-59-1	--	--	--	--	24	100
Naphthalene	91-20-3	--	--	--	--	23	100
Nitrobenzene	98-95-3	--	--	--	--	33	100
N-Nitrosodimethylamine	62-75-9	--	--	--	--	33	100
N-Nitroso-Di-n-Propylamine	621-64-7	--	--	--	--	26	100
N-nitrosodiphenylamine	86-30-6	--	--	--	--	17	100
Pentachloronitrobenzene	82-68-8	--	--	--	--	24	100
Pentachlorophenol	87-86-5	--	--	--	--	86	501
Phenanthrene	85-01-8	--	--	--	--	25	100
Phenol	108-95-2	--	--	--	--	24	100
Pyrene	129-00-0	--	--	--	--	23	100
Pyridine	110-86-1	--	--	--	--	101	501

PALs are not applicable to free product (NAPL) or Investigation Derived Waste.

QAPP WORKSHEET #15-T: REFERENCE LIMITS AND EVALUATION TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix	NAPL/IDW
Analytical Group	Pesticides via EPA Method 8081A (Fixed Lab)
Concentration Level	High

Analyte	CAS Number	Project Action Limit (mg/kg)			Project Quantitation Limit (mg/kg)	Achievable Laboratory Limits (mg/kg)	
		None	None	None		MDL/LOD	RL/LOQ
4,4´-DDD	72-54-8	--	--	--	--	0.32	1.00
4,4´-DDE	72-55-9	--	--	--	--	0.32	1.00
4,4´-DDT	50-29-3	--	--	--	--	0.32	1.00
Aldrin	309-00-2	--	--	--	--	0.32	1.00
alpha-BHC	319-84-6	--	--	--	--	0.32	1.00
alpha-Chlordane	5103-71-9	--	--	--	--	0.32	1.00
beta-BHC	319-85-7	--	--	--	--	0.32	1.00
Chlordane (Total)	57-74-9	--	--	--	--	3.56	12.0
delta-BHC	319-86-8	--	--	--	--	0.32	1.00
Dieldrin	60-57-1	--	--	--	--	0.32	1.00
Endosulfan I	959-98-8	--	--	--	--	0.32	1.00
Endosulfan II	33213-65-9	--	--	--	--	0.32	1.00
Endosulfan Sulfate	1031-07-8	--	--	--	--	0.32	1.00
Endrin	72-20-8	--	--	--	--	0.32	1.00
Endrin Aldehyde	7421-93-4	--	--	--	--	0.32	1.00
Endrin Ketone	53494-70-5	--	--	--	--	0.32	1.00
gamma-BHC (Lindane)	58-89-9	--	--	--	--	0.32	0.60
gamma-Chlordane	5566-34-7	--	--	--	--	0.32	1.00
Heptachlor	76-44-8	--	--	--	--	0.32	1.00
Heptachlor Epoxide	1024-57-3	--	--	--	--	0.32	1.00
Hexachlorobenzene	118-74-1	--	--	--	--	0.32	1.00
Methoxychlor	72-43-5	--	--	--	--	0.32	1.00
Toxaphene	8001-35-2	--	--	--	--	5.84	50.0

QAPP WORKSHEET #15-U: REFERENCE LIMITS AND EVALUATION TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix	NAPL/IDW
Analytical Group	PCBs via EPA Method 5035/8081A (Fixed Lab)
Concentration Level	High

Analyte	CAS Number	Project Action Limit (mg/kg)			Project Quantitation Limit (mg/kg)	Achievable Laboratory Limits (mg/kg)	
		None	None	None		MDL/LOD	RL/LOQ
Aroclor 1016	12674-11-2	--	--	--	--	0.5	1.0
Aroclor 1221	11104-28-2	--	--	--	--	0.5	1.0
Aroclor 1232	11141-16-5	--	--	--	--	0.5	1.0
Aroclor 1242	53469-21-9	--	--	--	--	0.5	1.0
Aroclor 1248	12672-29-6	--	--	--	--	0.5	1.0
Aroclor 1254	11097-69-1	--	--	--	--	0.5	1.0
Aroclor 1260	11096-82-5	--	--	--	--	0.5	1.0
Aroclor 1262	37324-23-5	--	--	--	--	0.5	1.0
Aroclor 1268	11100-14-4	--	--	--	--	0.5	1.0

PALs are not applicable to free product (NAPL) or Investigation Derived Waste.

QAPP WORKSHEET #15-V: REFERENCE LIMITS AND EVALUATION TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix	NAPL/IDW
Analytical Group	TAL Metals via EPA Method 6010/7470/7000 (Fixed Lab)
Concentration Level	High

Analyte	CAS Number	Project Action Limit (mg/kg)			Project Quantitation Limit (mg/kg)	Achievable Laboratory Limits (mg/kg)	
		None	None	None		MDL/LOD	RL/LOQ
Aluminum	7429-90-5	--	--	--	--	13.0	50.0
Antimony	7440-36-0	--	--	--	--	0.6	2.5
Arsenic	7440-38-2	--	--	--	--	0.4	2.5
Barium	7440-39-3	--	--	--	--	1.5	25.0
Beryllium	7440-41-7	--	--	--	--	0.1	0.5
Cadmium	7440-43-9	--	--	--	--	0.5	2.5
Calcium	7440-70-2	--	--	--	--	32.5	100
Chromium	7440-47-3	--	--	--	--	1.5	10.0
Cobalt	7440-48-4.	--	--	--	--	1.0	10.0
Copper	7440-50-8	--	--	--	--	2.0	10.0
Iron	7439-89-6	--	--	--	--	11.5	50.0
Lead	7439-92-1	--	--	--	--	2.5	10.0
Magnesium	7439-95-4	--	--	--	--	21.0	100.0
Manganese	7439-96-5	--	--	--	--	3.0	10.0
Mercury	7439-97-6	--	--	--	--	0.12	0.50
Nickel	7440-02-0	--	--	--	--	1.0	25.0
Potassium	7440-09-7	--	--	--	--	55.0	2500
Selenium	7782-49-2	--	--	--	--	8.5	25.0
Silver	7440-22-4	--	--	--	--	1.0	5.0
Sodium	7646-69-7	--	--	--	--	135.0	2500
Thallium	7440-28-0	--	--	--	--	0.4	1.5
Vanadium	7440-62-2	--	--	--	--	1.0	10.0
Zinc	7440-66-6	--	--	--	--	4.5	10.0

PALs are not applicable to free product (NAPL) or Investigation Derived Waste.

QAPP WORKSHEET #15-W: REFERENCE LIMITS AND EVALUATION TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix	NAPL/IDW
Analytical Group	TPH & Petroleum fingerprint via EPA Method 8100M/8015M (Fixed Lab)
Concentration Level	na

Analyte	CAS Number	Project Action Limit (mg/kg)				Project Quantitation Limit (mg/kg)	Achievable Laboratory Limits (mg/kg)	
		None	None	None	None		MDL/LOD	RL/LOQ
TPH	NA	--	--	--	--	--	5.6	37.5
Petroleum fingerprint	NA	--	--	--	--	--	NA	

PALs are not applicable to free product (NAPL) or Investigation Derived Waste.

NA = Not applicable.

QAPP WORKSHEET #15-X: REFERENCE LIMITS AND EVALUATION TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix	NAPL/IDW
Analytical Group	Viscosity via ASTM D445-06 (@40°C) (Fixed Lab)
Concentration Level	High

Analyte	CAS Number	Project Action Limit (mg/kg)			Project Quantitation Limit (mg/kg)	Achievable Laboratory Limits (mg/kg)	
		None	None	None		MDL/LOD	RL/LOQ
Viscosity	NA	--	--	--	--	NA	NA

PALs are not applicable to free product (NAPL) or Investigation Derived Waste.

NA = Not applicable.

QAPP WORKSHEET #15-Y: REFERENCE LIMITS AND EVALUATION TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix	NAPL/IDW
Analytical Group	Specific Gravity via ASTM D287-92 (Fixed Lab)
Concentration Level	High

Analyte	CAS Number	Project Action Limit (mg/kg)			Project Quantitation Limit (mg/kg)	Achievable Laboratory Limits (mg/kg)	
		None	None	None		MDL/LOD	RL/LOQ
Specific Gravity	NA	--	--	--	--	NA	NA

PALs are not applicable to free product (NAPL) or Investigation Derived Waste.

NA = Not applicable.

QAPP WORKSHEET #16: PROJECT SCHEDULE TIMELINE TABLE

☐ Worksheet Not Applicable (State Reason)

		Dates (MM/DD/YY)			
Activities	**Organization**	**Anticipated Date(s) of Initiation**	**Anticipated Date(s) of Completion**	**Deliverable**	**Deliverable Due Date**
Pore Water, Surface Water, Sediment Sampling	Stone	July 2013	August 2013	*FS Report*	November 2013
Rock Sampling	Stone	July 2013	August 2013	*FS Report*	November 2013
Soil Sampling	Stone	June 2013	July 2013	*FS Report*	November 2013
Groundwater Sampling	Stone	July 2013	September 2013	*FS Report*	November 2013
NAPL Sampling	Stone	July 2013	August 2013	*FS Report*	November 2013
Residential Well Sampling	Stone	July 2013	September 2013	*FS Report*	November 2013

QAPP WORKSHEET #17: SAMPLING DESIGN AND RATIONALE

☐ Worksheet Not Applicable (State Reason)

Refer to the Section 2 of the *SAP*, the *Field Sampling Plan*, for details.

Groundwater Monitoring Activities: Collect groundwater samples from up to 50 groundwater monitoring wells for one or more of the following reasons:

- Obtain representative total and dissolved metals samples from approximately 8 shallow overburden monitoring wells. These samples will be analyzed for total and dissolved TAL metals (Al, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Ma, Hg, Ni, K, Se, Sb, Na, Ag, Th, V, Zn) by USEPA Methods 6010/7470/7000;
- Obtain additional VOC results from select monitoring wells to support trend analysis. These samples will be analyzed for VOCs via USEPA Method 8260B;
- Obtain representative total and dissolved metals samples from approximately 18 overburden and bedrock monitoring wells located south and east of the Site, to determine if the dissolved VOC plume is affecting metals concentrations in groundwater. These samples will be analyzed for total and dissolved TAL metals by USEPA Methods 6010/7470/7000;and/or
- Obtain current geochemical data to support the evaluation of natural attenuation in the dissolved groundwater plumes. These samples will be field screened for nitrate/nitrite, sulfate/sulfite, ferrous/ferric iron, and chloride using a Hach field test kit, and analyzed by a fixed lab for VOCs via USEPA Method 8260B, ethane/ethane via Method RSK 175, total organic carbon (TOC) via USEPA Method 9060A, and *Dehalcoccoides* spp. (qDHC). In addition, should the qDHC analyses indicate that microbial degradation is occurring, additional analyses for TCE reductase (tceA), vinyl chloride reductase (bvcA), vinyl chloride reductase (vcrA) by proprietary methods will be performed.

Pore, Surface Water, and Sediment Assessment: Collect pore water, surface water, and sediment samples from the Hall Creek system for VOC analysis to use in ecological and human health risk evaluations and to support the assessment of remedial alternatives in the FS. Samples to be collected from twelve locations previously sampled by The Johnson Company (in 2010) following a record precipitation event, which may have resulted in non-representative analytical results. In addition, a 13th location in the northern wetland, will be accessed via a Fletcher Road property, and a staff gauge will be installed. Samples will be analyzed for VOCs by EPA Method 8260B, and select samples will be analyzed for total and dissolved TAL metals by USEPA Methods 6010/7470/7000.

Rock Core Assessment: Collect 12 pre-existing rock core samples for porosity analysis to evaluate the potential for matrix diffusion to play a significant role in source persistence and the likely performance of source-area remedial measures.

Source Area Investigation: Collect soil samples from 13 initial soil borings advanced in the western portion of the Source Area (vicinity of groundwater monitoring well EA-102D) to verify the presence or absence of NAPL and, if present, identify the physical and chemical properties of the NAPL and delineate its horizontal and vertical distribution. Data will also be used to support the update of the human health and ecological risk assessments. Collect soil samples from an additional 15 soil borings advanced in the remainder of the Source Area to document current conditions and delineate the extent of 10 mg/Kg total CVOC concentrations. Additional soil boring locations will be selected based on the results of the initial near-real time soil analytical results. An estimated 17 soil samples will be collected from each soil core and analyzed by the onsite laboratory for VOCs by Method 8260C. Additional samples will be collected from the remainder of the Source Area, to provide source characterization data for the FS. Aliquots of 10 selected soil samples will be further analyzed by a fixed laboratory for TPH. Sample selection will be biased towards soils that exhibit the highest PID readings and or visual/olfactory evidence of contamination within the "smear zone". In addition, 10 selected soil samples (five from the sand/gravel unit and five from the basal till unit) will be collected for analysis of the

following physical properties: bulk density and porosity, and fraction organic carbon (FOC).

If NAPL is detected in the western portion of the Source Area, up to three NAPL collection wells will be installed. If mobile NAPL accumulates in the wells, up to three NAPL samples will be collected to identify the physical and chemical properties of the NAPL and delineate its horizontal and vertical distribution

Shallow Groundwater Investigation, North Kingstown DPW Property: Two soil borings will be advanced at the North Kingstown DPW Property for the installation of shallow overburden groundwater monitoring wells. Soil cores from the North Kingstown DPW Property will be field screened for VOCs and a soil sample from each boring will be submitted for laboratory analysis for and analyzed by the onsite laboratory for VOCs. Groundwater samples will be collected and analyzed for VOCs.

Residential Well Water: Collect well water samples from 2 wells to be analyzed for VOCs by EPA Method 524.2 to allow the project team to make decisions regarding potential impact to drinking water receptors. Locations comprise 710 and 772 Fletcher Road.

QAPP WORKSHEET #18: SAMPLING LOCATIONS AND METHODS/SOP REQUIREMENTS TABLE

☐ Worksheet Not Applicable (State Reason)

Sampling Location/ID Number	Matrix	Depth (units)	Analytical Group	Concentration Level	Number of Samples (Identify Field Duplicates)	Sampling SOP Reference	Rationale for Sampling Location
BR-A BR-B BR-D BR-F EA-102D EA-104D EA-104R EA-106D EA-106R ME-1 ME-2 ME-3 ME-4 MW01-13D MW01-13R MW01-13S MW02-03D MW02-03R MW02-03S MW02-11D MW03-03D MW03-03R MW03-03S MW03-11R MW03-11S NMW-1 NMW-2 NMW-3 NMW-4 OB-AS OB-AD OB-CS OB-CD OB-DS OB-DD	Groundwater	varies	VOCs	Low	Fixed Lab:53 samples, 3 dup, 4/4 FB/TB, 3 MS/MSD	SEI SOP 6.27.3, 15a	To determine the nature and extent of VOCs in groundwater
			Total Metals		Fixed Lab: 26 samples, 2 dup, 2 FB, 2 MS/MSD		To determine the nature and extent of total metals in groundwater
			Dissolved Metals		Fixed Lab: 26 samples, 2 dup, 2 FB, 2 MS/MSD		To determine the nature and extent of dissolved metals in groundwater
			Ethane/ethene		Fixed Lab: 51 samples, 3 dup, 4/4 FB/TB, 3 MS/MSD		To determine natural attenuation parameters
			TOC		Fixed Lab: 51 samples, 3 dup		
			qDHC, tceA, bvcA and vcrA		Fixed Lab: 10 samples, 1 dup		
			Nitrate/nitrite sulfate/sulfite ferrous/ferric iron chloride		Field: 51 samples, 3 dup, 3 MS/MSD		

Sampling Location/ID Number	Matrix	Depth (units)	Analytical Group	Concentration Level	Number of Samples (Identify Field Duplicates)	Sampling SOP Reference	Rationale for Sampling Location
OB-ED OB-FS OB-FD OB-GS OB-HS OB-HD OB-IS OB-ID SES-01D SES-01R SES-01S SES-09D SES-09R SES-09S							
SEI3-9.0-NAPL through SEI5-9.0-NAPL	NAPL/IDW	9 ft (estimated)	VOCs	High	Fixed Lab: 3 samples, 1TB	SEI SOP 6.47.0, 23a	To determine NAPL characteristics
			SVOCs		Fixed Lab: 3 samples		
			TPH		Fixed Lab: 3 samples		
			Pesticides		Fixed Lab: 3 samples		
			PCBs		Fixed Lab: 3 samples		
			Fuel Fingerprint		Fixed Lab: 3 samples		
			Viscosity		Fixed Lab: 3 samples		
			Specific Gravity		Fixed Lab: 3 samples		
SEI1-9.0- GW and SEI2-9.0- GW	Soil	7 ft (estimated)	VOCs	Low	Mobile Lab: 2 samples, 0 dup, 1/1 FB/TB, 0 MS/MSD	SEI SOP 6.45.0, 21a	To assess soil vapor migration

Sampling Location/ID Number	Matrix	Depth (units)	Analytical Group	Concentration Level	Number of Samples (Identify Field Duplicates)	Sampling SOP Reference	Rationale for Sampling Location
	Groundwater	9 ft (estimated)	VOCs	Low	Fixed Lab: 2 samples, 1 dup, 1/1 FB/TB, 1 MS/MSD	SEI SOP 6.27.3, 15a	
WS-25 and WS-26	Well Water	shallow	VOCs	Low	Fixed Lab: 2 samples, 1 dup, 1/1 FB/TB, 1 MS/MSD	SEI SOP 6.44.0, 20a	Potential for migration and risk assessment
HC1-0.9-PW through HC12-0.9-PW, NW1-0.9-PW	Pore Water	20 cm below sediment surface	VOCs	Low	Fixed Lab: 13 samples, 1 dup, 1/1 TB/FB, 1 MS/MSD	SEI SOP 6.40.0, 18a	Potential for migration and risk assessment
			Total Metals		Fixed Lab: 7 samples, 1 dup, 1/0 FB, 1 MS/MSD		
			Dissolved Metals		Fixed Lab: 7 samples, 1 dup, 1/0 FB 1 MS/MSD		
HC1-0.9-SW through HC12-0.9-SW, NW1-0.9-SW	Surface Water	Surface water mid-depth (varies)	VOCs	Low	Fixed Lab: 13 samples, 1 dup, 1/1 TB/FB, 1 MS/MSD	SEI SOP 6.3.4, 8a	
			Total Metals		Fixed Lab: 7 samples, 1 dup, 1/0 FB/TB, 1 MS/MSD		
			Dissolved Metals		Fixed Lab: 7 samples, 1 dup, 1/0 FB/TB, 1 MS/MSD		
			Hardness		Fixed Lab: 13 samples, 1 dup, 1/0 FB/TB, 1 MS/MSD		
HC1-0.9-SD through HC12-0.9-SD, NW1-0.9-SD	Sediment	20 cm below sediment surface	VOCs	Low	Fixed Lab: 13 Samples, 1 dup, 1/1 FB/TB, 1 MS/MSD	SEI SOP 6.39.1, 17a	
			TOC		Fixed Lab: 13 Samples, 1 dup, FB		

Sampling Location/ID Number	Matrix	Depth (units)	Analytical Group	Concentration Level	Number of Samples (Identify Field Duplicates)	Sampling SOP Reference	Rationale for Sampling Location
SA1-0.0-SO through SA30-33.0-SO	Soil	0 to 35 ft bgs	VOCs	Low to High	Mobile Lab: 512 samples, 26 dup, 10/11 FB/TB, 26 MS/MSD	SEI SOP 6.45.0, 21a	Source Area characterization
			Porosity and Density		Fixed Lab: 10 samples	SEI SOP 6.45.0, 21a	
			FOC		Fixed Lab: 10 samples, 1 dup	SEI SOP 6.45.0, 21a	
			TPH		Fixed Lab: 10 samples, 1 dup, 1/1 FB/TB, 1 MS/MSD	SEI SOP 6.45.0, 21a	
Previously Cored Rock TBD	Rock	varies	Porosity	Low	Fixed Lab: 12 samples	SEI SOP 6.41.0, 19a	Source Area characterization

Specific analytical methods for groundwater samples are summarized in Table 3 of the SAP.

QAPP WORKSHEET #19: ANALYTICAL SOP REQUIREMENTS TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix	Analytical Group	Concentration Level	Analytical and Preparation Method/SOP Reference	Sample Volume	Containers (number, size and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/ analysis)
Groundwater	VOCs	Low	8260B/4b, 5b	120 mL	3 ×40-mL VOA vials	HCl, 4°C ±2°C	14 days
	Total Metals	Low	6010B/7470A/7000/ 6b, 7b, 8b, 9b	250 mL	1 × 250-mL polyethylene bottle	HNO_3, 4°C ±2°C	180 days
	Dissolved Metals	Low	6010B/7470A/7000/ 6b, 7b, 8b, 9b	250 mL	1 × 250-mL polyethylene bottle	HNO_3, 4°C ±2°C	180 days
	Ethane/ethene	Low	RSK 175/24b	80 mL	2 ×40-mL VOA vials	HCl, 4°C ±2°C	14 days
	TOC	Low	9060A/25b	80 mL	2 ×40-mL VOA vials	H_2SO_4, 4°C ±2°C	28 days
	qDHC, tceA, bvcA and vcrA	Low	Proprietary/28b	1 Liter	1 × 1-Liter polyethylene bottle	4°C ±2°C	2 days
	Nitrate, Nitrite, Sulfate, Sulfite, Ferrous iron, Ferric iron, Chloride	Low	Hach Field Methods, Spectrophotometric Nitrate – 8171 Nitrite – 8507 Sulfate – 8051 Sulfite – 8071 Ferrous iron – 8146 Total iron – 8008; Chloride – 8225/ 12b	TBD	TBD	TBD	TBD

Matrix	Analytical Group	Concentration Level	Analytical and Preparation Method/SOP Reference	Sample Volume	Containers (number, size and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/ analysis)
Surface Water	VOCs	Low	8260B/4b, 5b	120 mL	3 × 40-mL VOA vials	HCl, 4°C ±2°C	14 days
	Hardness	Low	6010A/9b	250 mL	1 × 250-mL polyethylene bottle	HNO_3, 4°C ±2°C	180 days
Pore Water	VOCs	Low	8260B/4b, 5b	120 mL	3 × 40-mL VOA vials	HCl, 4°C ±2°C	14 days
Well Water	VOCs	Low	524.2/13b	120 mL	3 × 40-mL VOA vials	HCl, 4°C ±2°C	14 days
Sediment	TOC	Low	Lloyd Kahn	100 grams	1 × 125-mL glass jar	4°C ±2°C	14 days
	VOCs	Low	8260B/4b, 5b	160 mL	3 × 40-mL VOA vials	1 × MeOH, 2 × DI water, 4°C ±2°C	14 days
Soil	VOCs	Low to High	8260C/27b	120 mL	2 × 40-mL VOA vials	1 × MeOH, 1 unpreserved, 4°C ±2°C	14 days
	Porosity & Density	Low to High	ASTM D7263/1b ASTM D1298/2b	200 grams	1 × 5-inch Macrocore® liner, sealed	4°C ±2°C	14 days
	FOC	Low to High	ASTM D 2974/3b	100 grams	1 × 125-mL glass jar	4°C ±2°C	14 days
	TPH	Low to High	8100M/22b	100 grams	1 × 125-mL glass jar	4°C ±2°C	14 days
Rock	Porosity	Low	API RP-40/29b	8 cubic inches	Polyethylene bag	none	none
NAPL	VOCs	High	8260B/4b, 4b	10 grams	1 × 40-mL VOA vial, (MeOH)	4°C ±2°C	14 days
	SVOCs	High	8270/23b	10 grams	1 × 40-mL VOA vial	4°C ±2°C	14 days

Matrix	Analytical Group	Concentration Level	Analytical and Preparation Method/SOP Reference	Sample Volume	Containers (number, size and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/ analysis)
	TPH/Fuel fingerprint	High	8100M/8015/22b	10 grams	1 × 40-mL VOA vial	4°C ±2°C	14 days
	Pesticides	High	8081A/20b	100 grams	1 × 125-mL glass jar	4°C ±2°C	14 days
	PCBs	High	8082/21b	10 grams	1 × 40-mL VOA vial	4°C ±2°C	1 year
	Viscosity	High	ASTM D 445-06 (@40°C)/26b	100 grams	125-mL glass jar	4°C ±2°C	14 days
	Specific Gravity	High	ASTM D445-06/ 14b	100 grams	125-mL glass jar	4°C ±2°C	14 days

QAPP WORKSHEET #20: FIELD QUALITY CONTROL SAMPLE SUMMARY TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix	Analytical Group	Concentration Level	Analytical and Preparation SOP Reference	No. of Sampling Locations	No. of Field Duplicate	No. of MS/MSD/ Dup	No. of Trip Blanks	No. of Equip. Blanks/Field Blank	No. of PT Samples	Total No. of Samples to Lab
Groundwater	VOCs	Low	8260B/4b, 5b	53	3	3	4	4	0	67
	Total Metals	Low	6010B/7470A/7000/ 6b, 7b, 8b, 9b	26	2	2	0	2	0	32
	Dissolved Metals	Low	6010B/7470A/7000/ 6b, 7b, 8b, 9b	26	2	2	0	2	0	32
	Ethane/ethene	Low	RSK 175/24b	51	3	3	4	4	0	65
	TOC	Low	9060A/25b	51	3	0	0	0	0	54
	qDHC, tceA, bvcA & vcrA	Low	Proprietary/28b	10	1	0	0	0	0	11
	Nitrate, Nitrite, Sulfate, Sulfite, Ferrous iron, Ferric iron, Chloride	Low	Hach Field Methods, Spectrophotometric: Nitrate – 8171; Nitrite – 8507; Sulfate – 8051; Sulfite – 8071; Ferrous iron – 8146; Total iron – 8008; Chloride – 8225 /12b	51	3	3	0	0	0	57
Well Water	VOCs	Low	524.2/13b	2	1	1	1	1	0	6
Pore Water	VOCs	Low	8260B/4b, 5b	13	1	1	1	1	0	17
	Total Metals	Low	6010B/7470A/7000/ 6b, 7b, 8b, 9b	7	1	1	0	1	0	10

Matrix	Analytical Group	Concentration Level	Analytical and Preparation SOP Reference	No. of Sampling Locations	No. of Field Duplicate	No. of MS/MSD/ Dup	No. of Trip Blanks	No. of Equip. Blanks/Field Blank	No. of PT Samples	Total No. of Samples to Lab
	Dissolved Metals	Low	6010B/7470A/7000/ 6b, 7b, 8b, 9b	7	1	1	0	1	0	10
Surface Water	VOCs	Low	8260B/4b, 5b	13	1	1	1	1	0	17
	Hardness	Low	6010	13	1	1	0	1	0	16
	Total Metals	Low	6010B/7470A/7000/ 6b, 7b, 8b, 9b	7	1	1	0	1	0	10
	Dissolved Metals	Low	6010B/7470A/7000/ 6b, 7b, 8b, 9b	7	1	1	0	1	0	10
Sediment	VOCs	Low	8260B/4b, 5b	13	1	1	1	1	0	17
	TOC	Low	Lloyd Kahn Method/25b	13	1	0	0	1	0	15
NAPL	VOCs	TBD	8260B/4b, 5b	3	0	0	1	0	0	4
	SVOCs	TBD	8270/23b	3	0	0	0	0	0	3
	TPH/ Petroleum fingerprint	TBD	8100M/8015M/22b	3	0	0	0	0	0	3
	PCBs	TBD	8082/21b	3	0	0	0	0	0	3
	Viscosity	TBD	ASTM D 445-06 (@40°C)/26b	3	0	0	0	0	0	3
	Specific Gravity	TBD	ASTM D287-92/	3	0	0	0	0	0	3
Soil	VOCs	TBD	8260C/27b	512	26	26	11	10	0	585
	Porosity & Density	TBD	ASTM D7263/1b	10	0	0	0	0	0	10
	FOC	TBD	ASTM D2974/3b	10	1	0	0	0	0	11

Matrix	Analytical Group	Concentration Level	Analytical and Preparation SOP Reference	No. of Sampling Locations	No. of Field Duplicate	No. of MS/MSD/ Dup	No. of Trip Blanks	No. of Equip. Blanks/Field Blank	No. of PT Samples	Total No. of Samples to Lab
	TPH	TBD	8100M/22b	10	1	1	1	1	0	14
Rock	Porosity	TBD	API RP 40/29b	12	0	0	0	0	0	12

QAPP WORKSHEET #21: PROJECT SAMPLING SOP REFERENCES TABLE

☐ Worksheet Not Applicable (State Reason)

Reference Number	Title, Revision Date and/or Number	Originating Organization	Equipment Type	Modified for Project Work?	Comments
1a	SOP SEI-4.2.7 Chain of Custody Procedures, 05/10/12, revision number: 7	Stone Environmental, Inc.	N/A	No	
2a	SOP SEI-4.5.11 Data Handling, Storage, Retrieval and Error Coding, 05/10/12, revision number: 11	Stone Environmental, Inc.	N/A	No	
3a	SOP SEI-5.1.5 Maintenance and Decontamination of Field Equipment, 02/20/04, revision number: 5	Stone Environmental, Inc.	N/A	No	
4a	SOP SEI-5.26.0 Use, Maintenance and Calibration of the LaMotte Model 2020*e* Turbidity Meter, 06/23/05, revision number: N/A	Stone Environmental, Inc.	Turbidity Meter	No	
5a	SOP SEI-5.27.1 Use, Maintenance and Calibration of the Hydrolab MS5 Water Quality Multiprobes, 07/12/12, revision number: 1	Stone Environmental, Inc.	Water Quality Sonde	No	
6a	SOP SEI-5.28.0 Use, Maintenance and Calibration of the Hach L Portable Dissolved Oxygen Meters (Hach Models HQ10 and HQ30d), 02/02/11, revision number: N/A	Stone Environmental, Inc.	Dissolved Oxygen Probe	No	
7a	SOP SEI-6.2.6 Water Level Measurement Use, Maintenance and Calibration of Electronic Water Level Indicators, 02/20/04, revision number: 6	Stone Environmental, Inc.	Water Level Indicators	No	
8a	SOP SEI-6.3.4 Surface Water Sampling, 02/24/04, revision number: 4	Stone Environmental, Inc.	N/A	No	
9a	SOP SEI-6.4.5 Installation, Development and Decommissioning of Monitoring Wells and Observation Wells, 08/01/07, revision number: 5	Stone Environmental, Inc.	N/A	No	
10a	SOP SEI-6.16.4 Handling, Collection and Transportation of Samples, 04/16/04, revision number: 4	Stone Environmental, Inc.	N/A	No	

Reference Number	Title, Revision Date and/or Number	Originating Organization	Equipment Type	Modified for Project Work?	Comments
11a	Hager-Richter Geoscience, Inc. *Standard Operating Procedures Utility Clearance for Boring Installations.*	Hager-Richter Geoscience, Inc.	Geonics EM-61-MK2, Sensors and Software Noggin Plus digital GPR system	No	
12a	SOP SEI-6.19.2 Use, Maintenance and Calibration of the Ionscience Phocheck 1000+ Photionization Detector (PID), 02/04/11, revision number: 2	Stone Environmental, Inc.	PID	No	
13a	SOP SEI-6.20.3 Undisturbed Soil Sample Collection Using a Thin Walled (Shelby) Tube, 11/29/05, revision number: 3	Stone Environmental, Inc.	Drill rig	No	
14a	SOP SEI-6.23.1 Observation and Monitoring well Surveying for Elevations, 11/29/05, revision number: 1	Stone Environmental, Inc.	Auto Level or Total Station	No	
15a	SOP SEI-6.27.3 Groundwater Sampling of Monitoring Wells, 11/29/05, revision number: 3	Stone Environmental, Inc.	Various Sampling Pumps	No	
16a	SOP SEI-6.34.0 Procedure for Sampling Groundwater Monitoring Wells Using Low Stress (Low Flow) Technique, 01/21/05, revision number: N/A	Stone Environmental, Inc.	Various Sampling Pumps	No	
17a	SOP SEI-6.39.1 Surface Sediment Sampling sing an EKMAN Bottom Grab Sampler, 10/16/12, revision number: 1	Stone Environmental, Inc.	EKMAN Grab Sampler	Yes	
18a	SOP SEI-40.0 Sediment Pore Water Sampling Using a (Henry's) Push Point (PP) Sampler, 08/21/12, revision number: N/A	Stone Environmental, Inc.	Henry's Push Point Sampler	Yes	
19a	SOP SEI-6.41.0 Hydrophobic Dye Testing for Light or Dense Non-Aqueous Phase Liquids (NAPL) Contamination, 08/21/12, revision number: N/A	Stone Environmental, Inc.	Oil-Red-O	Yes	
20a	SOP SEI-6.44.0 Procedure for Sample Collection From Residences and Commercial (Potable) Drinking Water Supplies, 10/15/12, revision: NA	Stone Environmental, Inc.	N/A	Yes	

Reference Number	Title, Revision Date and/or Number	Originating Organization	Equipment Type	Modified for Project Work?	Comments
21a	SOP SEI-6.45.0 Collection, Handling, and Preservation of Discrete Soil Samples, 10/15/12, revision: NA	Stone Environmental, Inc.	N/A	Yes	
22a	SOP SEI-6.46.0 Geologic Description of Unconsolidated Deposits Using the Unified Soil Classification System, 10/15/12, revision: NA	Stone Environmental, Inc.	N/A	Yes	
23a	SOP SEI-6.47.0 Non-Aqueous Phase Liquid (NAPL) Sampling, 10/16/12, revision: NA	Stone Environmental, Inc.	N/A	Yes	
24a	SOP SEI-6.42.2 Geologic Description of Unconsolidated Deposits. 10/16/12, revision: 2	Stone Environmental, Inc.	N/A	Yes	

QAPP WORKSHEET #22: FIELD EQUIPMENT CALIBRATION, MAINTENANCE, TESTING AND INSPECTION TABLE

☐ Worksheet Not Applicable (State Reason)

Field Equipment	Calibration Activity	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference[1]
Ionscience Phocheck 1000+ Photionization Detector	Calibrate according to manufacturer's specification	Maintain according to manufacturer's specifications	Check using calibration standards at mid-day	Inspect unit for damage	Daily	± 3%	Manufacturer service or replace unit	Field staff	12a
LaMotte Model 2020e Turbidimeter	Calibrate according to manufacturer's specification	Maintain according to manufacturer's specifications	Check using calibration standards at mid-day	Inspect unit for damage	Daily	± 3%	Manufacturer service or replace unit	Field staff	4a
Hydrolab MS5 Water Quality Multiprobes	Calibrate according to manufacturer's specification	Maintain according to manufacturer's specifications	Check using calibration standards at mid-day	Inspect unit for damage	Daily	± 3%	Manufacturer service or replace unit	Field staff	5a
Hach L Portable Dissolved Oxygen Meter, Models HQ10 and HQ30d	Calibrate according to manufacturer's specification	Maintain according to manufacturer's specifications	Check using calibration standards at mid-day	Inspect unit for damage	Daily	± 3%	Manufacturer service or replace unit	Field staff	6a
Water level Indicators	Calibrate according to manufacturer's specification	Maintain according to manufacturer's specifications	Check using calibrated bucket at mid-day	Inspect unit for damage	Daily	± 3%	Manufacturer service or replace unit	Field staff	7a

[1]From the Project Sampling SOP Reference Table (Worksheet #21)

QAPP WORKSHEET #23: ANALYTICAL SOP REFERENCES TABLE

☐ Worksheet Not Applicable (State Reason)

Reference Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work?
1b	SOP GT3.0 Standard Operating Procedure for Total Unit Weight (Bulk Density), 09/12/12, revision number: 1	Definitive	Bulk Density	NA	ESS Laboratory	No
2b	SOP GT5.0 Standard Operating Procedure for Standard Test Method for Specific Gravity of Soils, 09/12/12, revision number: 1	Definitive	Soil Porosity	NA	ESS Laboratory	No
3b	SOP GT7.0 Standard Operating Procedure for Moisture, Ash and Organic Matter of Peat and Other Organic Soils, 09/12/12, revision number: 1	Definitive	FOC	NA	ESS Laboratory	No
4b	SOP NO. 20_5035 Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS): Capillary Column Technique (SW-846 Method 5035), 03/13/09, revision number: 9	Definitive	VOCs	NA	ESS Laboratory	No
5b	SOP NO. 20_8260B Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS): Capillary Column Technique (EPA Method 624/SW-846 Method 8260B), 03/12/09, revision number: 10	Definitive	VOCs	Gas Chromatograph/ Mass Spectrometer (GC/MS)	ESS Laboratory	No
6b	SOP NO. 30_2009 Determination of Trace Elements by Stabilized Temperature Graphite Furnace Atomic Absorption (Methods EPA 200.9. 279.2, SM 3113B 20th Ed, MCP CAM III and SW846 7000 Series, 07/12/10, revision number: 8	Definitive	Metals	Graphite Furnace/ Atomic Absorption (GFAA)	ESS Laboratory	No

Reference Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work?
7b	SOP NO.30_2451 Mercury Analysis for Aqueous Samples, 03/24/05, revision number 4	Definitive	Metals	Cold Vapor Atomic Absorption (CVAA)	ESS Laboratory	No
8b	SOP NO. 30_3005 Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by GFAA, FLAA or ICP Spectroscopy (EPA 200.7 PREP/SW846 3005A/ SW846 3020A), 06/30/10, revision number: 5	Definitive	Metals	NA	ESS Laboratory	No
9b	SOP NO. 30_6010 Inductively Coupled Plasma-Atomic Emission Spectroscopy (SW 846 Method 6010B,C/ EPA Method 200.7/MCP-CAM IIIA), 07/15/10, revision number: 13	Definitive	Metals	Inductively Coupled Plasma Atomic Emission Spectrometer (ICP)	ESS Laboratory	No
10b	reserved					
11b	reserved					
12b	Hach *Water Analyses Handbook, 5th Edition*, 2008.	Definitive	Natural Attenuation Parameters	Hach Spectrophotometer	Stone	No
13b	SOP No. 20.0002A, Measurement of Purgeable Organic Compounds in Water by Capillary Column Gas Chromatography/Mess Spectometry (EPA Method 524.2)	Definitive	VOCs	GC/MS	ESS Laboratory	No
14b	ASTM D287-92, Standard Test Method for API Gravity of Crude Petroleum and Petroleum Products, (Hydrometer Method)	Definitive	Specific Gravity	NA	ESS Laboratory	No
15b	SOP NO. 40_0025I Anions by Ion Chromatography (Method EPA 300.0/SW846 9056A), 04/15/08, revision number: 3	Definitive	Hardness	Ion Chromatograph (IC)	ESS Laboratory	No

Reference Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work?
16b	SOP NO. 50_3510C Separatory Funnel Liquid-Liquid Extraction (SW-846 Method 3510C), 01/09/07, revision number: 10	Definitive		Separatory Funnel	ESS Laboratory	No
17b	SOP NO. 50_3540C Soxhlet Extraction (SW-846 Method 3540C), 05/01/07, revision number: 3	Definitive		Soxhlet	ESS Laboratory	No
18B	SOP NO. 50_3541 Automated Soxhlet Extraction (SW-846 Method 3541), 08/13/07, revision number: 3	Definitive		Soxhlet	ESS Laboratory	No
19b	SOP NO. 50_3546 Microwave Extraction (SW-846 Method 3546), 08/14/07, revision number: 1	Definitive		Microwave	ESS Laboratory	No
20b	SOP NO. 60_8081 Organochlorine Pesticides by Gas Chromatography: Capillary Column Technique (EPA Method 608/SW-846 Method 8081A and B/SM6630 C), 06/22/10, revision number: 6	Definitive	Pesticides	GC	ESS Laboratory	No
21b	SOP NO. 60_8082 Organochlorine PCB's as Aroclors by Gas Chromatography: Capillary Column Technique (EPA Method 608/SW-846 Method 8082), 04/27/07, revision number: 5	Definitive	Pesticides	GC	ESS Laboratory	No
22b	SOP NO. 60_8100-mod Total Petroleum Hydrocarbons (SW846 Method 8100 modified/Conn ETPH) by GC/FID, 06/23/08, revision number: 4	Definitive	TPH	GC/Flame Ionization Detector	ESS Laboratory	No
23b	SOP NO. 60_8270 Semivolatile Organic Compounds Analysis by Gas Chromatography/Mass Spectrometry (EPA Method 625/SW-846 Method 8270C and D), 08/17/10, revision number: 7	Definitive	SVOCs	GC/MS	ESS Laboratory	No

Reference Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work?
24b	SOP NO. 60_RSK175 Methane, Ethane and Ethene Gases in Water Samples by Gas Chromatography Analysis of Sample Headspace, 05/12/08, revision number: 1	Definitive	Ethane/Ethene	GC	ESS Laboratory	No
25b	SOP NO. BR-WC-002 Total Organic Carbon (Combustion Method) SM5310B (19th Edition) & SW-846 9060A, 04/01/11, revision number: 13	Definitive	TOC	IC	Test America	No
26b	ASTM Standard D 445-06 Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity), revision number: 6	Definitive	NAPL	NA	ESS Laboratory	No
27b	SEI-10.15.9 "The Determination of Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) (SW846 USEPA Method 8260C), 7 November 2012.	Definitive	VOCs	GC/MS	Stone	Yes
28b	Proprietary analytical methods for Dehalcoccoides spp. (qDHC), TCE reductase (tceA), vinyl chloride reductase (bvcA), vinyl chloride reductase (vcrA)	Definitive	Microbial	NA	Microbial Insights	No
29b	American Petroleum Institute, Recommended Practices for Core Analysis, Recommended Practice 40, Second Edition, February 1998.	Definitive	Porosity	NA	Core Laboratory	No

QAPP WORKSHEET #24: ANALYTICAL INSTRUMENT CALIBRATION TABLE

☐ Worksheet Not Applicable (State Reason)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
GC/FID	Initial calibration	Initial start of analysis	5- 7 point calibration with RSD < 20% , coefficient must be 0.995 or higher using linear fit.	Establish new calibration curve or service instrument	Laurel Stoddard	22b
	Continuing calibration verification	Every 10 samples	Result of continuing calibration verification must be within ±20%, or separate continuing calibration verification prepared from second source must be ±20%.	Verify solution integrity and check instrument performance. Perform necessary maintenance and recalibrate instrument. Reanalyze all affected samples		
GC/MS	Initial calibration	Initial start of analysis	5-7 point correlation or a correlation factor (r) of 0.99 for linear regression	Establish new calibration curve or service instrument	Michael Rossi/ Laurel Stoddard	27b, 4b, 5b, 23b
	Continuing calibration verification	Daily (every 12 hours) .	Minimum RRF and Maximum % D of ±20%, according to the laboratory SOP.	Verify solution integrity and check instrument performance. Perform necessary maintenance and recalibrate instrument. Reanalyze all affected samples		

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
GF/AA	Initial calibration	Daily or at start of run as required in the laboratory SOP	3-point correlation coefficient must be 0.998 or higher using a linear fit; additional calibration verifications per control limits provided in SOP.	Establish new calibration curve or service instrument	Laurel Stoddard	8b
	Continuing calibration verification	Every 10 samples	See laboratory SOP	Verify solution integrity and check instrument performance. Perform necessary maintenance and recalibrate instrument. Reanalyze all affected samples		
	End-of-run calibration verification	Every 10 samples	End-of-run calibration verification	Verify solution integrity and check instrument performance. Perform necessary maintenance and recalibrate instrument. Reanalyze all affected samples		
CVAA	Initial calibration	Daily or at start of run as required in the laboratory SOP	7-point calibration with correlation coefficient of 0.995 or higher using a linear fit; additional calibration verifications per control limits in SOP.	Establish new calibration curve or service instrument	Laurel Stoddard	7b

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
	Continuing calibration verification	Every 10 samples and end of run	Results of continuing calibration verification must be ±20% of expected values, result of continuing calibration blank must be below PQL.	Terminate analysis, correct problem, reanalyze previous 10 samples		
ICP/AES	Initial calibration	Daily or at start of run	3-point correlation coefficient must be 0.998 or higher using a linear fit; additional calibration verifications per control limits provided in SOP.	Establish new calibration curve or service instrument	Laurel Stoddard	9b
	Continuing calibration verification	Every 10 samples	Continuing calibration verification	Continuing calibration verification		
	End-of-run calibration verification	Every 10 samples	End-of-run calibration verification	End-of-run calibration verification		
Hach Spectrophotometer	Continuing calibration verification	Every 10 samples	Result of continuing calibration verification must be within ±20%, or separate continuing calibration verification prepared from second source must be ±20%.	Recalibrate and reanalyze samples	Michael Rossi	12b

QAPP WORKSHEET #25: ANALYTICAL INSTRUMENT AND EQUIPMENT MAINTENANCE, TESTING AND INSPECTION TABLE

☐ Worksheet Not Applicable (State Reason)

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference[1]
GC/FID	According to manufacturer's instructions						Laurel Stoddard	22b
GC/MS	According to manufacturer's instructions						Michael Rossi/ Laurel Stoddard	27b, 4b, 5b, 23b
GF/AA	According to manufacturer's instructions						Laurel Stoddard	8b
CVAA	According to manufacturer's instructions						Laurel Stoddard	7b
ICP/AES	According to manufacturer's instructions						Laurel Stoddard	9b
Hach Spectrophotometer	According to manufacturer's instructions						Michael Rossi	12b

[1] From the Analytical SOP References table (Worksheet #23)

QAPP WORKSHEET #26: SAMPLE HANDLING SYSTEM

☐ Worksheet Not Applicable (State Reason)

Task	
SAMPLE COLLECTION, PACKAGING, AND SHIPMENT	
Sample Collection:	Stone field staff
Sample Packaging:	Stone field staff
Coordination of Shipment:	Stone field staff
Type of Shipment/Carrier:	Coolers, UPS or FedEx overnight
Sample Receipt, Custody and Storage, Preparation, Determinative Analysis	SEI, ESS Laboratory, Core Laboratory, Microbial Insights
SAMPLE ARCHIVING	
Field Sample Storage	N/A, Stone Mobile laboratory
Sample Extract/Digestate storage	See Worksheet #19 for holding times
Biological Sample Storage	N/A
Personnel/Organization	Fixed Facilities Responsibility
SAMPLE DISPOSAL	
Personnel/Organization	SEI, ESS Laboratory, Core Laboratory, Microbial Insights

QAPP WORKSHEET #27: SAMPLE CUSTODY REQUIREMENTS

☐ Worksheet Not Applicable (State Reason)

Field Sample Custody Procedures (sample collection, packaging, shipment and delivery): All samples will be placed in appropriate containers according to Worksheet #19. Sample labels will include: site name, date, time, preservative, and analytical method. Preprinted labels provided by Stone Environmental, Inc. will be used as much as possible. All samples will be documented on a Chain of Custody, which will accompany the samples to the laboratory. Except geotechnical soil samples, which will be collected in buckets, all samples will be placed in coolers with wet ice and maintained at 4°C ± 2°C during storage and shipping. Coolers will be packed with sufficient packing material (not Styrofoam) to minimize movement during transit. The coolers will be sealed with tape and shipped via UPS or FEDEX (overnight) to the appropriate laboratory. The laboratory will be responsible for notifying Stone Environmental, Inc. as soon as possible of any inconsistencies or breakage upon receipt. Trip Blanks will be prepared in the laboratory and Field blanks will be prepared in the field, where appropriate and shipped with the empty bottles to the Site. Samples of the Chain of Custody, custody tape, and labels are provided on the receiving laboratory.
Laboratory Sample Custody Procedures (receipt of samples, archiving, disposal): The laboratory will check all samples against the Chain of Custody upon receipt, and will electronically archive all samples before analysis. Samples will be disposed of by the laboratory in accordance with applicable RCRA Hazardous Waste regulations.
Sample Identification Procedures: The first set of characters will be the alphanumeric location ID which will identify the sample location. Samples from existing wells will use the most recent sample name for that well. All other locations will be assigned an individual consecutive number preceded by a two-letter code for the type of sample. Each location will be assigned an individual consecutive number preceded by a two letter code for the type of sample. The location ID will be followed by a dash. The second set of characters will identify the depth interval rounded to the nearest foot below ground surface. Surface waters will be measured below water surface, and where depth intervals are not valid an "X" will be used. The depth interval will be followed by a dash. The third set of characters will identify the type of sample taken ("GW" Groundwater sample, "SW" Surface water, "PW" Pore water, "SD" Sediment, "SO" Soil sample, "D" Field duplicate, "EB" Equipment blank, "TB" Trip Blank, "MS" Matrix spike sample, "MSD" Matrix spike duplicate sample, "IDW" Investigation Derived Waste sample. Full details are located in Subsection 2.9 of the SAP.

QAPP WORKSHEET #28-A: QC SAMPLES TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix	Soil	**Sampler's Name**	Field Staff
Analytical Group	VOCs	**Field Sampling Organization**	Stone
Sampling SOP	21a	**Analytical Organization**	Stone Mobile Lab
Analytical Method/ SOP Reference	EPA Method 8260C/ASTMD6520/27b	**No. of Sample Locations**	510

QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Duplicate sample	1 per 20 samples	RPD ≤ 50%	Reanalyze samples or review similarity of samples.	Michael Rossi	Precision/Bias-contamination	RPD ≤ 50%
Aqueous VOA Trip Blank	1 per cooler for VOC samples	No reported concentrations ≥ quantitation limits	Review storage and handling procedures; alter as necessary.	Michael Rossi	Contamination	No reported concentrations ≥ quantitation limits
Equipment/Field Blank	1 per event	No reported concentrations ≥ quantitation limits	Review equipment decontamination procedures; alter as necessary.	Michael Rossi	Contamination	No reported concentrations ≥ quantitation limits
Method Blank	1 per 12-hrs	No reported concentrations ≥ ½ quantitation limits	Redigest samples with new method blank and reanalyze for analyte.	Michael Rossi	Contamination	No reported concentrations ≥ quantitation limits
Lab Control Sample	1 per 12-hrs	DOD limits – see attached tables	Analyte may not be reported without a narrative; rerun batch.	Michael Rossi	Accuracy	DOD limits – see attached tables
Matrix Spike/Matrix Spike Duplicate	1 per 20 samples or one per matrix type	DOD limits – see attached tables; RPD ≤ 30%	Note deviation in report.	Michael Rossi	Sensitivity, Accuracy/precision bias	DOD limits – see attached tables; RPD ≤ 30%

QAPP WORKSHEET #28-B: QC SAMPLES TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix	Aqueous		**Sampler's Name**		Field Staff
Analytical Group	Natural Attenuation Parameters		**Field Sampling Organization**		Stone
Sampling SOP	16a		**Analytical Organization**		Stone Field
Analytical Method/ SOP Reference	Hach Field Methods, Spectrophotometric/ 12b		**No. of Sample Locations**		50

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Duplicate sample	1 per 20 samples	RPD ≤ 30%	Reanalyze samples or review similarity of samples.	Michael Rossi	Accuracy/Bias-contamination	RPD ≤ 30%
Method Blank	1 per 12-hrs	No reported concentrations ≥ quantitation limits	Redigest samples with new method blank and reanalyze for analyte.	Michael Rossi	Accuracy	No reported concentrations ≥ quantitation limits
Lab Control Sample	1 per 12-hrs	±30% recovery	Analyte may not be reported without a narrative; rerun batch.	Michael Rossi	Accuracy	±30% recovery
Matrix Spike/Matrix Spike Duplicate	1 per 20 samples or one per matrix type	±30% recovery RPD ≤ 30%	Note deviation in report.	Michael Rossi	Sensitivity, Accuracy/precision bias	±30% recovery RPD ≤ 30%

QAPP WORKSHEET #28-C: QC SAMPLES TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix	Aqueous	**Sampler's Name**	Field Staff
Analytical Group	VOCs	**Field Sampling Organization**	Stone
Sampling SOP	16a	**Analytical Organization**	Fixed Lab
Analytical Method/ SOP Reference	EPA Method 8260B/5035/4b, 5b	**No. of Sample Locations**	52

QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Duplicate sample	1 per 20 samples	RPD ≤ 30%	Reanalyze samples or review similarity of samples.	Laurel Stoddard	Precision/Bias	RPD ≤ 30%
Aqueous VOA Trip Blank	1 per cooler for VOC samples	No reported concentrations ≥ quantitation limits	Review storage and handling procedures; alter as necessary.	Laurel Stoddard	Contamination	No reported concentrations ≥ quantitation limits
Equipment/Field Blank	1 per event	No reported concentrations ≥ quantitation limits	Review equipment decontamination procedures; alter as necessary.	Laurel Stoddard	Contamination	No reported concentrations ≥ quantitation limits
Method Blank	1 per 12-hrs	No reported concentrations ≥ ½ quantitation limits	Redigest samples with new method blank and reanalyze for analyte.	Laurel Stoddard	Contamination	No reported concentrations ≥ quantitation limits
Lab Control Sample	1 per 12-hrs	DOD limits – see attached tables	Analyte may not be reported without a narrative; rerun batch.	Laurel Stoddard	Accuracy/precsion	DOD limits – see attached tables
Matrix Spike/Matrix Spike Duplicate	1 per 20 samples or one per matrix type	DOD limits – see attached tables; RPD ≤ 30%	Note deviation in report.	Laurel Stoddard	Sensitivity, Accuracy/precision bias	DOD limits – see attached tables; RPD ≤ 30%

QAPP WORKSHEET #28-D: QC SAMPLES TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix	Aqueous	**Sampler's Name**	Field Staff
Analytical Group	TAL Metals	**Field Sampling Organization**	Stone
Sampling SOP	16a	**Analytical Organization**	Fixed Lab
Analytical Method/ SOP Reference	EPA Method 6010B/7470A/7000/6b, 7b, 8b, 9b	**No. of Sample Locations**	8

QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Duplicate sample	1 per 20 samples	RPD ≤ 30%	Reanalyze samples or review similarity of samples.	Laurel Stoddard	Precision/Bias-contamination	RPD ≤ 30%
Aqueous VOA Trip Blank	1 per cooler for VOC samples	No reported concentrations ≥ quantitation limits	Review storage and handling procedures; alter as necessary.	Laurel Stoddard	Contamination	No reported concentrations ≥ quantitation limits
Equipment/Field Blank	1 per event	No reported concentrations ≥ quantitation limits	Review equipment decontamination procedures; alter as necessary.	Laurel Stoddard	Contamination	No reported concentrations ≥ quantitation limits
Method Blank	1 per 20 samples	No reported concentrations ≥ ½ quantitation limits	Redigest samples with new method blank and reanalyze for analyte.	Laurel Stoddard	Accuracy	No reported concentrations ≥ quantitation limits
Lab Control Sample	1 per 20 samples	DOD limits – see attached tables	Analyte may not be reported without a narrative; rerun batch.	Laurel Stoddard	Accuracy/precision	DOD limits – see attached tables
Matrix Spike/Matrix Spike Duplicate	1 per 20 samples or one per matrix type	DOD limits – see attached tables; RPD ≤ 30%	Note deviation in report.	Laurel Stoddard	Sensitivity, Accuracy/precision bias	DOD limits – see attached tables; RPD ≤ 30%

QAPP WORKSHEET #28-E: QC SAMPLES TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix	Aqueous	**Sampler's Name**	Field Staff
Analytical Group	Ethane/ethene	**Field Sampling Organization**	Stone
Sampling SOP	16a	**Analytical Organization**	Fixed Lab
Analytical Method/ SOP Reference	RSK 175/24b	**No. of Sample Locations**	50

QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Duplicate sample	1 per 20 samples	RPD ≤ 30%	Reanalyze samples or review similarity of samples.	Laurel Stoddard	Precision/Bias	RPD ≤ 30%
Equipment/Field Blank	1 per event	No reported concentrations ≥ quantitation limits	Review equipment decontamination procedures; alter as necessary.	Laurel Stoddard	Contamination	No reported concentrations ≥ quantitation limits
Method Blank	1 per 20 samples	No reported concentrations ≥ ½ quantitation limits	Redigest samples with new method blank and reanalyze for analyte.	Laurel Stoddard	Accuracy	No reported concentrations ≥ quantitation limits
Lab Control Sample	1 per 20 samples	Lab limits – see attached tables	Analyte may not be reported without a narrative; rerun batch.	Laurel Stoddard	Accuracy/precision	±40% recovery
Matrix Spike/Matrix Spike Duplicate	1 per 20 samples or one per matrix type	Lab limits – see attached tables; RPD ≤ 30%	Note deviation in report.	Laurel Stoddard	Sensitivity, Accuracy/precision bias	±40% recovery; RPD ≤ 30%

QAPP WORKSHEET #28-F: QC SAMPLES TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix	Aqueous	**Sampler's Name**	Field Staff
Analytical Group	TOC	**Field Sampling Organization**	Stone
Sampling SOP	16a	**Analytical Organization**	Fixed Lab
Analytical Method/ SOP Reference	EPA Method 9060A/25b	**No. of Sample Locations**	50

QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Duplicate sample	1 per 20 samples	RPD ≤ 30%	Reanalyze samples or review similarity of samples.	Laurel Stoddard	Precision/Bias	RPD ≤ 30%
Equipment/Field Blank	1 per event	No reported concentrations ≥ quantitation limits	Review equipment decontamination procedures; alter as necessary.	Laurel Stoddard	Contamination	No reported concentrations ≥ quantitation limits
Method Blank	1 per 20 samples	No reported concentrations ≥ ½ quantitation limits	Redigest samples with new method blank and reanalyze for analyte.	Laurel Stoddard	Contamination	No reported concentrations ≥ quantitation limits
Lab Control Sample	1 per 20 samples	Lab limits – see attached tables	Analyte may not be reported without a narrative; rerun batch.	Laurel Stoddard	Accuracy/precision	±40% recovery
Matrix Spike/Matrix Spike Duplicate	1 per 20 samples or one per matrix type	Lab limits – see attached tables; RPD ≤ 30%	Note deviation in report.	Laurel Stoddard	Sensitivity, Accuracy/precision bias	±15% recovery; RPD ≤ 20%

QAPP WORKSHEET #28-G: QC SAMPLES TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix	reserved			**Sampler's Name**		
Analytical Group				**Field Sampling Organization**		
Sampling SOP				**Analytical Organization**		
Analytical Method/ SOP Reference				**No. of Sample Locations**		
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Duplicate sample					Precision/Bias	
Equipment/Field Blank					Contamination	
Method Blank					Contamination	
Lab Control Sample					Accuracy/precision	
Matrix Spike/Matrix Spike Duplicate					Sensitivity, Accuracy/precision bias	

QAPP WORKSHEET #28-H: QC SAMPLES TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix	reserved			**Sampler's Name**		
Analytical Group				**Field Sampling Organization**		
Sampling SOP				**Analytical Organization**		
Analytical Method/ SOP Reference				**No. of Sample Locations**		
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Duplicate sample					Precision/Bias	
Equipment/Field Blank					Contamination	
Method Blank					Contamination	
Lab Control Sample					Accuracy/precision	
Matrix Spike/Matrix Spike Duplicate					Sensitivity, Accuracy/precision bias	

QAPP WORKSHEET #28-I: QC SAMPLES TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix	Aqueous	**Sampler's Name**	Field Staff
Analytical Group	qDHC, tceA, bvcA and vcrA	**Field Sampling Organization**	Stone
Sampling SOP	16a	**Analytical Organization**	Fixed Lab
Analytical Method/ SOP Reference	Proprietary/28b	**No. of Sample Locations**	50

QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Duplicate sample	1 per 20 samples	RPD ≤ 30%	Reanalyze samples or review similarity of samples.	Jennifer Ericson	Precision/Bias	RPD ≤ 30%
Equipment/Field Blank	1 per event	TBD	Review equipment decontamination procedures; alter as necessary.	Jennifer Ericson	Contamination	TBD
Method Blank	1 per 20 samples	No reported concentrations ≥ ½ quantitation limits	Redigest samples with new method blank and reanalyze for analyte.	Jennifer Ericson	Contamination	No reported concentrations ≥ quantitation limits
Lab Control Sample	1 per 20 samples	TBD	Analyte may not be reported without a narrative; rerun batch.	Jennifer Ericson	Accuracy/precision	TBD

QAPP WORKSHEET #28-J: QC SAMPLES TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix	Aqueous	**Sampler's Name**	Field Staff
Analytical Group	Hardness	**Field Sampling Organization**	Stone
Sampling SOP	8a	**Analytical Organization**	Fixed Lab
Analytical Method/ SOP Reference	EPA Method 6010B/9b	**No. of Sample Locations**	TBD

QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Duplicate sample	1 per 20 samples	RPD ≤ 30%	Reanalyze samples or review similarity of samples.	Laurel Stoddard	Precision/Bias	RPD ≤ 30%
Equipment/Field Blank	1 per event	No reported concentrations ≥ quantitation limits	Review equipment decontamination procedures; alter as necessary.	Laurel Stoddard	Contamination	No reported concentrations ≥ quantitation limits
Method Blank	1 per 20 samples	No reported concentrations ≥ ½ quantitation limits	Redigest samples with new method blank and reanalyze for analyte.	Laurel Stoddard	Contamination	No reported concentrations ≥ quantitation limits
Lab Control Sample	1 per 20 samples	Lab limits – see attached tables	Analyte may not be reported without a narrative; rerun batch.	Laurel Stoddard	Accuracy/precision	Lab limits – see attached tables
Matrix Spike/Matrix Spike Duplicate	1 per 20 samples or one per matrix type	Lab limits – see attached tables; RPD ≤ 30%	Note deviation in report.	Laurel Stoddard	Sensitivity, Accuracy/precision bias	Lab limits – see attached tables; RPD ≤ 30%

QAPP WORKSHEET #28-K: QC SAMPLES TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix	Aqueous	**Sampler's Name**	Field Staff
Analytical Group	VOCs	**Field Sampling Organization**	Stone
Sampling SOP	20a	**Analytical Organization**	Fixed Lab
Analytical Method/ SOP Reference	EPA Method 524.2/13b	**No. of Sample Locations**	52

QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Duplicate sample	1 per 20 samples	RPD ≤ 30%	Reanalyze samples or review similarity of samples.	Laurel Stoddard	Precision/Bias	RPD ≤ 30%
Aqueous VOA Trip Blank	1 per cooler for VOC samples	No reported concentrations ≥ quantitation limits	Review storage and handling procedures; alter as necessary.	Laurel Stoddard	Contamination	No reported concentrations ≥ quantitation limits
Equipment/Field Blank	1 per event	No reported concentrations ≥ quantitation limits	Review equipment decontamination procedures; alter as necessary.	Laurel Stoddard	Contamination	No reported concentrations ≥ quantitation limits
Method Blank	1 per 20 samples	No reported concentrations ≥ ½ quantitation limits	Redigest samples with new method blank and reanalyze for analyte.	Laurel Stoddard	Contamination	No reported concentrations ≥ quantitation limits
Lab Control Sample	1 per 20 samples	Lab limits – see attached tables	Analyte may not be reported without a narrative; rerun batch.	Laurel Stoddard	Accuracy/precision	Lab limits – see attached tables
Matrix Spike/Matrix Spike Duplicate	1 per 20 samples or one per matrix type	Lab limits – see attached tables; RPD ≤ 30%	Note deviation in report.	Laurel Stoddard	Sensitivity, Accuracy/precision bias	Lab limits – see attached tables; RPD ≤ 30%

QAPP WORKSHEET #28-L: QC SAMPLES TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix	Soil (Sediment)	**Sampler's Name**	Field Staff
Analytical Group	VOCs	**Field Sampling Organization**	Stone
Sampling SOP	17a	**Analytical Organization**	Fixed Lab
Analytical Method/ SOP Reference	EPA Method 8260B/4b, 5b	**No. of Sample Locations**	52

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Duplicate sample	1 per 20 samples	RPD ≤ 50%	Reanalyze samples or review similarity of samples.	Laurel Stoddard	Precision/Bias	RPD ≤ 50%
Aqueous VOA Trip Blank	1 per cooler for VOC samples	No reported concentrations ≥ quantitation limits	Review storage and handling procedures; alter as necessary.	Laurel Stoddard	Contamination	No reported concentrations ≥ quantitation limits
Equipment/Field Blank	1 per event	No reported concentrations ≥ quantitation limits	Review equipment decontamination procedures; alter as necessary.	Laurel Stoddard	Contamination	No reported concentrations ≥ quantitation limits
Method Blank	1 per 12-hrs	No reported concentrations ≥ ½ quantitation limits	Redigest samples with new method blank and reanalyze for analyte.	Laurel Stoddard	Contamination	No reported concentrations ≥ quantitation limits
Lab Control Sample	1 per 12-hrs	Lab limits – see attached tables	Analyte may not be reported without a narrative; rerun batch.	Laurel Stoddard	Accuracy/precision	Lab limits – see attached tables
Matrix Spike/Matrix Spike Duplicate	1 per 20 samples or one per matrix type	Lab limits – see attached tables; RPD ≤ 30%	Note deviation in report.	Laurel Stoddard	Sensitivity, Accuracy/precision bias	Lab limits – see attached tables; RPD ≤ 30%

QAPP WORKSHEET #28-M: QC SAMPLES TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix	Soil (Sediment)			**Sampler's Name**		Field Staff
Analytical Group	TOC			**Field Sampling Organization**		Stone
Sampling SOP	17a			**Analytical Organization**		Fixed Lab
Analytical Method/ SOP Reference	Lloyd Kahn Method/ 25b			**No. of Sample Locations**		TBD
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Duplicate sample	1 per 20 samples	RPD ≤ 50%	Reanalyze samples or review similarity of samples.	Laurel Stoddard	Precision/Bias	RPD ≤ 50%
Equipment/Field Blank	1 per event	No reported concentrations ≥ quantitation limits	Review equipment decontamination procedures; alter as necessary.	Laurel Stoddard	Contamination	No reported concentrations ≥ quantitation limits
Method Blank	1 per 20 samples	No reported concentrations ≥ ½ quantitation limits	Redigest samples with new method blank and reanalyze for analyte.	Laurel Stoddard	Contamination	No reported concentrations ≥ quantitation limits
Lab Control Sample	1 per 20 samples	±30% recovery	Analyte may not be reported without a narrative; rerun batch.	Laurel Stoddard	Accuracy/precision	±30% recovery
Matrix Spike/Matrix Spike Duplicate	1 per 20 samples or one per matrix type	±30% recovery; RPD ≤ 30%	Note deviation in report.	Laurel Stoddard	Sensitivity, Accuracy/precision bias	±30% recovery; RPD ≤ 30%

QAPP WORKSHEET #28-N: QC SAMPLES TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix		Soil		**Sampler's Name**		Field Staff
Analytical Group		Porosity & Density		**Field Sampling Organization**		Stone
Sampling SOP		17a		**Analytical Organization**		Fixed Lab
Analytical Method/ SOP Reference		ASTM D7263 & ASTM D1298/ 1b, 2b		**No. of Sample Locations**		TBD
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Duplicate sample	1 per 20 samples	RPD ≤ 50%	Reanalyze samples or review similarity of samples.	Laurel Stoddard	Precision/Bias	RPD ≤ 50%
Lab Duplicate	1 per 20 samples	±30% recovery	Analyte may not be reported without a narrative; rerun batch.	Laurel Stoddard	Precision/Bias	±30% recovery

QAPP WORKSHEET #28-O: QC SAMPLES TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix	Soil	**Sampler's Name**	Field Staff
Analytical Group	Fraction organic carbon	**Field Sampling Organization**	Stone
Sampling SOP	21a	**Analytical Organization**	Fixed Lab
Analytical Method/ SOP Reference	ASTM D2974/3b	**No. of Sample Locations**	10

QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Duplicate sample	1 per 20 samples	RPD ≤ 50%	Reanalyze samples or review similarity of samples.	Laurel Stoddard	Precision/Bias	RPD ≤ 50%
Equipment/Field Blank	1 per event	No reported concentrations ≥ quantitation limits	Review equipment decontamination procedures; alter as necessary.	Laurel Stoddard	Contamination	No reported concentrations ≥ quantitation limits
Method Blank	1 per 20 samples	No reported concentrations ≥ ½ quantitation limits	Redigest samples with new method blank and reanalyze for analyte.	Laurel Stoddard	Contamination	No reported concentrations ≥ quantitation limits
Lab Control Sample	1 per 20 samples	±30% recovery	Analyte may not be reported without a narrative; rerun batch.	Laurel Stoddard	Accuracy/precision	±30% recovery
Matrix Spike/Matrix Spike Duplicate	1 per 20 samples or one per matrix type	±30% recovery; RPD ≤ 30%	Note deviation in report.	Laurel Stoddard	Sensitivity, Accuracy/precision bias	±30% recovery; RPD ≤ 30%

QAPP WORKSHEET #28-P: QC SAMPLES TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix	Soil	**Sampler's Name**	Field Staff
Analytical Group	TPH	**Field Sampling Organization**	Stone
Sampling SOP	21a	**Analytical Organization**	Fixed Lab
Analytical Method/ SOP Reference	EPA Method 8100M/ 22b	**No. of Sample Locations**	10

QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Duplicate sample	1 per 20 samples	RPD ≤ 50%	Reanalyze samples or review similarity of samples.	Laurel Stoddard	Precision/Bias	RPD ≤ 50%
Equipment/Field Blank	1 per event	No reported concentrations ≥ quantitation limits	Review equipment decontamination procedures; alter as necessary.	Laurel Stoddard	Contamination	No reported concentrations ≥ quantitation limits
Method Blank	1 per 20 samples	No reported concentrations ≥ ½ quantitation limits	Redigest samples with new method blank and reanalyze for analyte.	Laurel Stoddard	Contamination	No reported concentrations ≥ quantitation limits
Lab Control Sample	1 per 20 samples	40-140% recovery	Analyte may not be reported without a narrative; rerun batch.	Laurel Stoddard	Accuracy/precision	40-140% recovery
Matrix Spike/Matrix Spike Duplicate	1 per 20 samples or one per matrix type	40-140% recovery; RPD ≤ 50%	Note deviation in report.	Laurel Stoddard	Sensitivity, Accuracy/precision bias	40-140% recovery; RPD ≤ 50%

QAPP WORKSHEET #28-Q: QC SAMPLES TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix	Rock	**Sampler's Name**	Field Staff
Analytical Group	Porosity	**Field Sampling Organization**	Stone
Sampling SOP	10a	**Analytical Organization**	Fixed Lab
Analytical Method/ SOP Reference	API RP-40/29b	**No. of Sample Locations**	TBD

QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Duplicate sample	1 per 20 samples	RPD ≤ 50%	Reanalyze samples or review similarity of samples.	Hugh Peek	Precision/Bias	RPD ≤ 50%
Lab Duplicate	1 per 20 samples	±30% recovery	Analyte may not be reported without a narrative; rerun batch.	Hugh Peek	Precision/Bias	±30% recovery

QAPP WORKSHEET #28-R: QC SAMPLES TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix	NAPL/IDW	**Sampler's Name**	Field Staff
Analytical Group	VOCs	**Field Sampling Organization**	Stone
Sampling SOP	23a	**Analytical Organization**	Fixed Lab
Analytical Method/ SOP Reference	EPA Method 8260B/4b, 5b	**No. of Sample Locations**	3

QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Duplicate sample	1 per 20 samples	RPD ≤ 50%	Reanalyze samples or review similarity of samples.	Laurel Stoddard	Precision/Bias	RPD ≤ 50%
Aqueous VOA Trip Blank	1 per cooler for VOC samples	No reported concentrations ≥ quantitation limits	Review storage and handling procedures; alter as necessary.	Laurel Stoddard	Contamination	No reported concentrations ≥ quantitation limits
Equipment/Field Blank	1 per event	No reported concentrations ≥ quantitation limits	Review equipment decontamination procedures; alter as necessary.	Laurel Stoddard	Contamination	No reported concentrations ≥ quantitation limits
Method Blank	1 per 12-hrs	No reported concentrations ≥ ½ quantitation limits	Redigest samples with new method blank and reanalyze for analyte.	Laurel Stoddard	Contamination	No reported concentrations ≥ quantitation limits
Lab Control Sample	1 per 12-hrs	DOD limits – see attached tables	Analyte may not be reported without a narrative; rerun batch.	Laurel Stoddard	Accuracy/precision	DOD limits – see attached tables
Matrix Spike/Matrix Spike Duplicate	1 per 20 samples or one per matrix type	DOD limits – see attached tables; RPD ≤ 30%	Note deviation in report.	Laurel Stoddard	Sensitivity, Accuracy/precision bias	DOD limits – see attached tables; RPD ≤ 30%

QAPP WORKSHEET #28-S: QC SAMPLES TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix	NAPL/IDW	**Sampler's Name**	Field Staff
Analytical Group	SVOCs	**Field Sampling Organization**	Stone
Sampling SOP	23a	**Analytical Organization**	Fixed Lab
Analytical Method/ SOP Reference	EPA Method 8270/23b	**No. of Sample Locations**	3

QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Duplicate sample	1 per 20 samples	RPD ≤ 50%	Reanalyze samples or review similarity of samples.	Laurel Stoddard	Precision/Bias	RPD ≤ 50%
Equipment/Field Blank	1 per event	No reported concentrations ≥ quantitation limits	Review equipment decontamination procedures; alter as necessary.	Laurel Stoddard	Contamination	No reported concentrations ≥ quantitation limits
Method Blank	1 per 20 samples	No reported concentrations ≥ ½ quantitation limits	Redigest samples with new method blank and reanalyze for analyte.	Laurel Stoddard	Contamination	No reported concentrations ≥ quantitation limits
Lab Control Sample	1 per 20 samples	DOD limits – see attached tables	Analyte may not be reported without a narrative; rerun batch.	Laurel Stoddard	Accuracy/precision	DOD limits – see attached tables
Matrix Spike/Matrix Spike Duplicate	1 per 20 samples or one per matrix type	DOD limits – see attached tables; RPD ≤ 30%	Note deviation in report.	Laurel Stoddard	Sensitivity, Accuracy/precision bias	DOD limits – see attached tables; RPD ≤ 30%

QAPP WORKSHEET #28-T: QC SAMPLES TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix	NAPL/IDW	**Sampler's Name**	Field Staff
Analytical Group	Pesticides	**Field Sampling Organization**	Stone
Sampling SOP	23a	**Analytical Organization**	Fixed Lab
Analytical Method/ SOP Reference	EPA Method 8081A/20b	**No. of Sample Locations**	3

QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Duplicate sample	1 per 20 samples	RPD ≤ 50%	Reanalyze samples or review similarity of samples.	Laurel Stoddard	Precision/Bias-contamination	RPD ≤ 50%
Equipment/Field Blank	1 per event	No reported concentrations ≥ quantitation limits	Review equipment decontamination procedures; alter as necessary.	Laurel Stoddard	Contamination	No reported concentrations ≥ quantitation limits
Method Blank	1 per 20 samples	No reported concentrations ≥ ½ quantitation limits	Redigest samples with new method blank and reanalyze for analyte.	Laurel Stoddard	Contamination	No reported concentrations ≥ quantitation limits
Lab Control Sample	1 per 20 samples	Lab limits – see attached tables	Analyte may not be reported without a narrative; rerun batch.	Laurel Stoddard	Accuracy/precision	Lab limits – see attached tables
Matrix Spike/Matrix Spike Duplicate	1 per 20 samples or one per matrix type	Lab limits – see attached tables; RPD ≤ 30%	Note deviation in report.	Laurel Stoddard	Sensitivity, Accuracy/precision bias	Lab limits – see attached tables; RPD ≤ 30%

QAPP WORKSHEET #28-U: QC SAMPLES TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix		NAPL/IDW		**Sampler's Name**		Field Staff
Analytical Group		PCBs		**Field Sampling Organization**		Stone
Sampling SOP		23a		**Analytical Organization**		Fixed Lab
Analytical Method/ SOP Reference		EPA Method 8082/21b		**No. of Sample Locations**		3
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Duplicate sample	1 per 20 samples	RPD ≤ 50%	Reanalyze samples or review similarity of samples.	Laurel Stoddard	Precision/Bias	RPD ≤ 50%
Equipment/Field Blank	1 per event	No reported concentrations ≥ quantitation limits	Review equipment decontamination procedures; alter as necessary.	Laurel Stoddard	Contamination	No reported concentrations ≥ quantitation limits
Method Blank	1 per 20 samples	No reported concentrations ≥ ½ quantitation limits	Redigest samples with new method blank and reanalyze for analyte.	Laurel Stoddard	Contamination	No reported concentrations ≥ quantitation limits
Lab Control Sample	1 per 20 samples	Lab limits – see attached tables	Analyte may not be reported without a narrative; rerun batch.	Laurel Stoddard	Accuracy/precision	Lab limits – see attached tables
Matrix Spike/Matrix Spike Duplicate	1 per 20 samples or one per matrix type	Lab limits – see attached tables; RPD ≤ 30%	Note deviation in report.	Laurel Stoddard	Sensitivity, Accuracy/precision bias	Lab limits – see attached tables; RPD ≤ 30%

QAPP WORKSHEET #28-V: QC SAMPLES TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix	NAPL/IDW	**Sampler's Name**	Field Staff
Analytical Group	TAL Metals	**Field Sampling Organization**	Stone
Sampling SOP	23a	**Analytical Organization**	Fixed Lab
Analytical Method/ SOP Reference	EPA Method 6010B/7470A/7000/ 6b, 7b, 8b, 9b	**No. of Sample Locations**	3

QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Duplicate sample	1 per 20 samples	RPD ≤ 50%	Reanalyze samples or review similarity of samples.	Laurel Stoddard	Precision/Bias	RPD ≤ 50%
Equipment/Field Blank	1 per event	No reported concentrations ≥ quantitation limits	Review equipment decontamination procedures; alter as necessary.	Laurel Stoddard	Contamination	No reported concentrations ≥ quantitation limits
Method Blank	1 per 20 samples	No reported concentrations ≥ ½ quantitation limits	Redigest samples with new method blank and reanalyze for analyte.	Laurel Stoddard	Contamination	No reported concentrations ≥ quantitation limits
Lab Control Sample	1 per 20 samples	Lab limits – see attached tables	Analyte may not be reported without a narrative; rerun batch.	Laurel Stoddard	Accuracy/precision	Lab limits – see attached tables
Matrix Spike/Matrix Spike Duplicate	1 per 20 samples or one per matrix type	Lab limits – see attached tables; RPD ≤ 30%	Note deviation in report.	Laurel Stoddard	Sensitivity, Accuracy/precision bias	Lab limits – see attached tables; RPD ≤ 30%

QAPP WORKSHEET #28-W: QC SAMPLES TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix	NAPL/IDW	**Sampler's Name**	Field Staff
Analytical Group	TPH & Petroleum fingerprint	**Field Sampling Organization**	Stone
Sampling SOP	21a	**Analytical Organization**	Fixed Lab
Analytical Method/SOP Reference	EPA Method 8100M/8015M/ 22b	**No. of Sample Locations**	3

QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Duplicate sample	1 per 20 samples	RPD ≤ 50%	Reanalyze samples or review similarity of samples.	Laurel Stoddard	Precision/Bias	RPD ≤ 50%
Equipment/Field Blank	1 per event	No reported concentrations ≥ quantitation limits	Review equipment decontamination procedures; alter as necessary.	Laurel Stoddard	Contamination	No reported concentrations ≥ quantitation limits
Method Blank	1 per 20 samples	No reported concentrations ≥ ½ quantitation limits	Redigest samples with new method blank and reanalyze for analyte.	Laurel Stoddard	Contamination	No reported concentrations ≥ quantitation limits
Lab Control Sample	1 per 20 samples	40-140% recovery	Analyte may not be reported without a narrative; rerun batch.	Laurel Stoddard	Accuracy/precision	40-140% recovery
Matrix Spike/Matrix Spike Duplicate	1 per 20 samples or one per matrix type	40-140% recovery; RPD ≤ 50%	Note deviation in report.	Laurel Stoddard	Sensitivity, Accuracy/precision bias	40-140% recovery; RPD ≤ 50%

QAPP WORKSHEET #28-X: QC SAMPLES TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix	NAPL/IDW	**Sampler's Name**	Field Staff
Analytical Group	Viscosity	**Field Sampling Organization**	Stone
Sampling SOP	23a	**Analytical Organization**	Fixed Lab
Analytical Method/SOP Reference	ASTM D455-06, 26b	**No. of Sample Locations**	3

QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Duplicate sample	1 per 20 samples	RPD ≤ 50%	Reanalyze samples or review similarity of samples.	Laurel Stoddard	Precision/Bia	RPD ≤ 50%
Reference Sample (if applicable)	1 per 20 samples	RPD ≤ 30%	Analyte may not be reported without a narrative; rerun batch.	Laurel Stoddard	Accuracy	RPD ≤ 30%
Lab Duplicate	1 per 20 samples or one per matrix type	RPD ≤ 20%	Note deviation in report.	Laurel Stoddard	Precision/bias	RPD ≤ 20%

QAPP WORKSHEET #28-Y: QC SAMPLES TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix		NAPL/IDW		**Sampler's Name**		Field Staff
Analytical Group		Specific Gravity		**Field Sampling Organization**		Stone
Sampling SOP		23a		**Analytical Organization**		Fixed Lab
Analytical Method/SOP Reference		ASTM D287-92/14b		**No. of Sample Locations**		3
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Duplicate sample	1 per 20 samples	RPD ≤ 50%	Reanalyze samples or review similarity of samples.	Laurel Stoddard	Precision/Bias	RPD ≤ 50%
Reference Sample (if applicable)	1 per 20 samples	RPD ≤ 30%	Analyte may not be reported without a narrative; rerun batch.	Laurel Stoddard	Accuracy	RPD ≤ 30%
Lab Duplicate	1 per 20 samples or one per matrix type	RPD ≤ 20%	Note deviation in report.	Laurel Stoddard	Precision/Bias	RPD ≤ 20%

QAPP WORKSHEET #29: PROJECT DOCUMENTS AND RECORDS TABLE

☐ Worksheet Not Applicable (State Reason)

Sample Collection Documents and Records	On-Site Analysis Documents and Records	Off-Site Analysis Documents and Records	Data Assessment Documents and Records	Other
Field Notes	Sample receipt, custody, and tracking records	Sample receipt, custody, and tracking records	Field sampling audit checklist	
Chain of Custody Records	Equipment calibration logs	Equipment calibration logs	Field analysis audit checklist	
Air Bills	Sample collection logs	Samples collection logs	Data review and assessment reports	
Custody Seals	Equipment maintenance, testing and inspection logs	Equipment maintenance, testing and inspection logs	Corrective Action forms	
Telephone logs	Corrective action forms	Corrective action forms	Telephone logs	
Corrective action forms	Field screening result logs	Field screening result logs		
	Reported sample results	Reported sample results		
	Telephone logs	Reported results for standards, QC checks, and QC samples		
		Raw data for samples, standards, QC checks, and QC samples		
		Data package completeness checklist		
		Sample disposal records		
		Telephone logs		
		Extraction/cleanup records		
		Raw data (stored digitally)		

QAPP WORKSHEET #30: ANALYTICAL SERVICES TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix	Analytical Group	Concentration Level	Analytical SOP	Data Package Turnaround Time	Laboratory/ Organization	Backup Laboratory/ Organization
Soil	VOCs	Low to High	27b	24 hrs	Stone Environmental, Inc. 535 Stone Cutters Way Montpelier, VT 05602 Mike Rossi 802-229-2196	NA
		Low	4b, 5b	28 days	ESS Laboratory 185 Frances Avenue Cranston, RI 02910 Laurel Stoddard 401-461-7181	NA
	TPH	Low	22b	28 days		NA
	Porosity & Density	Not applicable	1b	28 days		NA
	FOC	Not applicable	3b	28 days		NA
	TOC	Not applicable	3b	28 days		NA
Aqueous	Nitrate, Nitrite, Sulfate, Sulfite, Ferrous iron, Ferric iron Chloride	Low	12b	24 hrs	Stone Environmental, Inc. (see above)	NA
	VOCs	Low	4b, 5b	28 days	ESS Laboratory (see above)	NA
	TAL Metals	Low	6b, 7b, 8b, 9b	28 days		NA
	Ethane/Ethene	Low	24b	28 days		NA
	Hardness	Low	15b	28 days		NA
	TOC	Low	3b, 25b	28 days		NA

	qDHC, tceA, bvcA and vcrA	Low	28b	10 days	Microbial Insights 2340 Stock Creek Blvd. Rockford, TN 37853-3044 865-573-8188	NA
NAPL	VOCs	High	4b, 5b	28 days	ESS Laboratory (see above)	NA
	SVOCs	High	23b	28 days		NA
	TPH	High	22b	28 days		NA
	Pesticides	High	20b, 23b	28 days		NA
	PCBs	High	21b	28 days		NA
	Petroleum Fingerprint	High	22b	28 days		NA
	Viscosity	High	26b	28 days		NA
	Specific Gravity	High	14b	28 days		NA
Rock	Porosity	Not applicable	29b	28 days	Core Laboratory 6316 Windfern Road Houston, TX 77040 713-328-3578	NA

QAPP WORKSHEET #31: PLANNED PROJECT ASSESSMENT TABLE

☐ Worksheet Not Applicable (State Reason)

Assessment Type	Frequency	Internal or External	Organization Performing Assessment	Person(s) Responsible for Performing Assessment (Title and Organizational Affiliation)	Person(s) Responsible for Responding To Assessment Findings (Title and Organizational Affiliation)	Person(s) Responsible for Identifying and Implementing Corrective Actions (CA) (Title and Organizational Affiliation)	Person(s) Responsible for Monitoring Effectiveness of CA (Title and Organizational Affiliation)
Field Sampling Technical Audit	1/At sampling startup	Internal	Stone Environmental, Inc.	Kim Watson, QAO, Stone	Joseph Schmidl, Project Manager, Stone Environmental, Inc.	Joseph Schmidl, Project Manager, Stone Environmental, Inc.	Kim Watson, QAO, Stone Environmental, Inc.
Laboratory Data Audit/Assessment	1/At receipt of sampling results	External	Stone Environmental, Inc.	Kim Watson, QAO, Stone	Laurel Stoddard, ESS Laboratory OR Mike Rossi, Stone Environmental, Inc.	Laurel Stoddard, ESS Laboratory OR Mike Rossi, Stone Environmental, Inc.	Kim Watson, QAO, Stone Environmental, Inc.

QAPP WORKSHEET #32: ASSESSMENT FINDINGS AND CORRECTIVE ACTION RESPONSES

☐ Worksheet Not Applicable (State Reason)

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings (Name, Title, Organization)	Timeframe of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response (Name, Title, Org.)	Timeframe for Response
Field Sampling Audit	Written Audit Report	Field Operations Leader (FOL) and Joseph Schmidl, Project Manager, Stone Environmental, Inc.	24 hours after audit	Letter or memorandum	Joseph Schmidl, Project Manager, Stone Environmental, Inc.	24 hours after notification
Laboratory Data Audit	Verbal report to laboratory, written report for internal documentation	Laurel Stoddard, ESS Laboratory OR Kim Watson, Stone Environmental, Inc.	24 hours after audit	Verbal report to laboratory, written report for internal documentation	Kim Watson, QAO, Stone Environmental, Inc.	24 hours after notification

QAPP WORKSHEET #33: QA MANAGEMENT REPORTS TABLE

☐ Worksheet Not Applicable (State Reason)

Type of Report	Frequency	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation (Title and Org.)	Report Recipient(s) (Title and Org.)
Field Sampling Audit	1/At startup of sampling	8/2013	Kim Watson, QAO, Stone Environmental, Inc.	FOL and Joseph Schmidl, Project Manager, Stone Environmental, Inc.
Data Usability Assessment Report	1/After all data are generated and checked	9/2013	Kim Watson, QAO, Stone Environmental, Inc.	Joseph Schmidl, Project Manager, Stone Environmental, Inc.
Final Project Report	1/After QA Management Reports and risk assessment completed	11/2013	Joseph Schmidl, Project Manager, Stone Environmental, Inc.	Casey Haskell, P.G., Project Manager, NAE; Joseph Schmidl, Project Manager, Stone Environmental, Inc.

QAPP WORKSHEET #34: VERIFICATION (STEP I) PROCESS TABLE

☐ Worksheet Not Applicable (State Reason)

Verification Input	Description	Internal/External	Responsible for Verification (Name, Organization)
Chain of custody and shipping forms	Chain of custody forms and shipping documentation will be reviewed internally upon their completion and verified against the packed sample coolers they represent. A copy of the chain of custody will be retained at the file site, and the original and remaining copies placed in a Ziploc bag inside the cooler for shipment. See chain of custody SOP for further details.	Internal	Joseph Schmidl, Project Manager, Stone Environmental, Inc.
Audit reports	Upon report completion, a copy of all audit reports will be saved in the site file. If corrective actions are required, a copy of the documented corrective action taken will be attached to the appropriate audit report in the site file. At the beginning of additional sampling and at the completion of site work, site file audit reports will be reviewed internally to ensure that all appropriate corrective actions have been taken and that corrective action reports are attached. If corrective actions have not been taken, the sampling team leader will be notified to ensure corrective action is taken.	Internal	Kim Watson, QAO, Stone Environmental, Inc.
Field Notes	Field notes will be reviewed internally and placed in the site file. A copy of the field notes will be attached to the final report.	Internal	Joseph Schmidl, Project Manager, Stone Environmental, Inc.
Laboratory Data	All laboratory data packages will be verified internally by the laboratory performing the work for completeness and technical accuracy prior to submittal. All received data packages will be verified internally according to the data validation procedures specified in Worksheet #34.	Internal Internal	Mike Rossi, Laboratory Director, Stone Environmental, Inc. Laurel Stoddard, ESS Laboratory. Kim Watson, QAO, Stone Environmental, Inc.

QAPP WORKSHEET #35: VALIDATION (STEPS IIA AND IIB) PROCESS TABLE

☐ Worksheet Not Applicable (State Reason)

Step IIa/IIb	Validation Input	Description	Responsible for Validation (Name, Organization)
IIa	Onsite analytical work and fixed laboratory work.	All onsite analytical data will be reviewed against QAPP requirements for completeness and accuracy based on the field calibration records.	Kim Watson, QAO, and Joseph Schmidl, Project Manager, Stone Environmental, Inc.
IIa	SOPs	Ensure that all sampling and analytical SOPs were followed.	Kim Watson, QAO, and Joseph Schmidl, Project Manager Stone Environmental, Inc.
IIa	Documentation of Method QC Results	Establish that all method required QC samples were run and met required limits.	Kim Watson, QAO, Stone Environmental, Inc.
IIa	Documentation of QAPP QC sample results.	Establish that all QAPP required QC samples were run and met required limits.	Kim Watson, QAO, Stone Environmental, Inc.
IIa	Project Quantitation Limits	All sample results met the project quantitation limits specified in the QAPP.	Kim Watson, QAO, and Joseph Schmidl, Project Manager Stone Environmental, Inc.

To the extent possible, Stone's validation will be performed in conformance with Tier II (Stage 2a electronic and manual) guidelines as defined by EPA Region I, Region I EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, March 1996; Region I EPA-NE Inorganic Data Validation Functional Guidelines, November 2008; Pesticide/PCB Data Validation Functional Guidelines (02/2004) - PART III. EPA's National Functional Guidelines for Organic Data Review, October 1999 and EPA's National Functional Guidelines for Inorganic Data Review, October 2004 were also considered during the evaluation, and professional judgment was applied as necessary and appropriate.

QAPP WORKSHEET #36: VALIDATION (STEPS IIA AND IIB) SUMMARY TABLE

☐ Worksheet Not Applicable (State Reason)

Step IIa/IIb	Matrix	Analytical Group	Concentration Level	Validation Criteria	Data Validator (Title and Org.)
IIa	Aqueous	VOCs, TAL Metals, Ethane/ethane, Hardness, TOC, qDHC, tceA, bvcA, and vcrA	Low	QAPP Worksheets #12-B to –K;15- B to –K ; and 28-B to –K	Kim Watson, QAO, Stone Environmental, Inc.
IIa	NAPL/IDW	VOCs, SVOCs, TPH, Pesticides, PCBs, Petroleum Fingerprint, Viscosity, Specific Gravity	High	QAPP Worksheets #12-R to –Y;15-R to –Y ; and 28-R to –Y	Kim Watson, QAO, Stone Environmental, Inc.
IIa	Soil	VOCs, TPH, Porosity, Density, FOC, TOC	Low to High	QAPP Worksheets #12-A, 12-L to –P;15-A, 15-L to –P ; and 28-A, 28-L to –P	Kim Watson, QAO, Stone Environmental, Inc.
IIa	Rock	Porosity	Not applicable	QAPP Worksheets #12-Q, 15-Q and 28-Q	Kim Watson, QAO, Stone Environmental, Inc.

QAPP WORKSHEET #37: USABILITY ASSESSMENT

☐ Worksheet Not Applicable (State Reason)

The Data Usability Assessment will be headed by Kim Watson, QAO, of Stone Environmental, Inc. The Project Manager will be responsible for assigning task work to the individual task members who will be supporting the Data Usability Assessment. After the Data Usability Assessment has been performed, data deemed appropriate for use will then be used to support the FS. The results of the Data Usability Assessment will be presented in the Final FS Report. The accuracy, precision and completeness of data will be assessed and conclusions drawn based on their results. Accuracy and precision of analytical results will be evaluated based on criteria specified in the analytical methods used. Such criteria will include, but not be limited to, blank results, matrix spike recoveries, surrogate percent recovery values, correlation coefficients, and detection limits.

Accuracy: The amount of agreement between the true value of a parameter and the measured value. Accuracy is a statistical measurement of correctness and includes components of random error (variability due to imprecision) and systemic error. It therefore reflects the total error associated with a measurement. A measurement is accurate when the value reported does not differ from the true value or known concentration of the spike or standard. Analytical accuracy is measured by comparing the percent recovery of analytes spiked into an LCS to a control limit. For volatile and semi-volatile organic compounds, surrogate compound recoveries are used to assess accuracy and method performance for each sample analyzed. The percent recovery will be calculated. The control limits for percent recovery of spike compounds and surrogates are listed in the laboratory SOP corresponding to the specific analytical method used for analysis.

Precision: The measurement of the agreement between samples from the same population. It is strictly defined as the degree of mutual agreement among independent measurements as the result of repeated application of the same process under similar conditions. Precision can be expressed as the standard deviation between independent samples or as the relative percent difference between duplicate samples.

Completeness: The measure of the amount of validated data obtained compared to that which was expected to be obtained. The number of valid results divided by the number of possible individual analyte results, expressed as a percentage, determines the completeness of a data set. For completeness requirements, valid results are all results not qualified with an "R" flag. An "R" flag is place on the data by the data validator and means that the data are unusable due to deficiencies in the ability to analyze the sample and meet QC criteria.

ATTACHMENT A

LIST OF METHOD PERFORMANCE CRITERIA FOR ALL ANALYTES

Project Accuracy and Precision Report

eQapp Name: Stone PR-58 02222013
Description:

Analyte Name	Analyte Label (CAS)	Lower Rejection (%)	Lower Recovery (%)	Upper Recovery (%)	Upper Rejection (%)	RPD (%)
Method: 5210B **Matrix: Water**						
QC Type: Laboratory_Control_Sample						
BIOLOGIC OXYGEN DEMAND, FIVE-DAY	BOD5	50.00	87.50	112.50		
Method: 5220D **Matrix: Water**						
QC Type: Laboratory_Control_Sample						
CHEMICAL OXYGEN DEMAND	COD	50.00	95.00	105.00		
QC Type: Matrix_Spike						
CHEMICAL OXYGEN DEMAND	COD	50.00	75.00	125.00		
Method: 524.2 **Matrix: Water**						
QC Type: Laboratory_Control_Sample						
1,1,1,2-TETRACHLOROETHANE	630-20-6	10.00	70.00	130.00		20.00
1,1,1-TRICHLOROETHANE	71-55-6	10.00	70.00	130.00		20.00
1,1,2,2-TETRACHLOROETHANE	79-34-5	10.00	70.00	130.00		20.00
1,1,2-TRICHLOROETHANE	79-00-5	10.00	70.00	130.00		20.00
1,1-DICHLOROETHANE	75-34-3	10.00	70.00	130.00		20.00
1,1-DICHLOROETHENE	75-35-4	10.00	70.00	130.00		20.00
1,1-DICHLOROPROPENE	563-58-6	10.00	70.00	130.00		20.00
1,2,3-TRICHLOROBENZENE	87-61-6	10.00	70.00	130.00		20.00
1,2,3-TRICHLOROPROPANE	96-18-4	10.00	70.00	130.00		20.00
1,2,4-TRICHLOROBENZENE	120-82-1	10.00	70.00	130.00		20.00
1,2,4-TRIMETHYLBENZENE	95-63-6	10.00	70.00	130.00		20.00
1,2-DIBROMO-3-CHLOROPROPANE	96-12-8	10.00	70.00	130.00		20.00
1,2-DIBROMOETHANE	106-93-4	10.00	70.00	130.00		20.00
1,2-DICHLOROBENZENE	95-50-1	10.00	70.00	130.00		20.00

Analyte Name	Analyte Label (CAS)	Lower Rejection (%)	Lower Recovery (%)	Upper Recovery (%)	Upper Rejection (%)	RPD (%)
Method: 524.2	**Matrix: Water**					
QC Type: Laboratory_Control_Sample						
1,2-DICHLOROETHANE	107-06-2	10.00	70.00	130.00		20.00
1,2-DICHLOROPROPANE	78-87-5	10.00	70.00	130.00		20.00
1,3,5-TRIMETHYLBENZENE	108-67-8	10.00	70.00	130.00		20.00
1,3-DICHLOROBENZENE	541-73-1	10.00	70.00	130.00		20.00
1,3-DICHLOROPROPANE	142-28-9	10.00	70.00	130.00		20.00
1,4-DICHLOROBENZENE	106-46-7	10.00	70.00	130.00		20.00
1,4-DIOXANE	123-91-1	10.00	70.00	130.00		20.00
2,2-DICHLOROPROPANE	594-20-7	10.00	70.00	130.00		20.00
2-BUTANONE	78-93-3	10.00	70.00	130.00		20.00
2-CHLOROTOLUENE	95-49-8	10.00	70.00	130.00		20.00
2-HEXANONE	591-78-6	10.00	70.00	130.00		20.00
4-CHLOROTOLUENE	106-43-4	10.00	70.00	130.00		20.00
4-METHYL-2-PENTANONE	108-10-1	10.00	70.00	130.00		20.00
ACETONE	67-64-1	10.00	70.00	130.00		20.00
BENZENE	71-43-2	10.00	70.00	130.00		20.00
BROMOBENZENE	108-86-1	10.00	70.00	130.00		20.00
BROMOCHLOROMETHANE	74-97-5	10.00	70.00	130.00		20.00
BROMODICHLOROMETHANE	75-27-4	10.00	70.00	130.00		20.00
BROMOFORM	75-25-2	10.00	70.00	130.00		20.00
BROMOMETHANE	74-83-9	10.00	70.00	130.00		20.00
CARBON DISULFIDE	75-15-0	10.00	70.00	130.00		20.00
CARBON TETRACHLORIDE	56-23-5	10.00	70.00	130.00		20.00
CHLOROBENZENE	108-90-7	10.00	70.00	130.00		20.00
CHLOROETHANE	75-00-3	10.00	70.00	130.00		20.00
CHLOROFORM	67-66-3	10.00	70.00	130.00		20.00
CHLOROMETHANE	74-87-3	10.00	70.00	130.00		20.00
CIS-1,2-DICHLOROETHENE	156-59-2	10.00	70.00	130.00		20.00
CIS-1,3-DICHLOROPROPENE	10061-01-5	10.00	70.00	130.00		20.00
DIBROMOCHLOROMETHANE	124-48-1	10.00	70.00	130.00		20.00
DIBROMOMETHANE	74-95-3	10.00	70.00	130.00		20.00
DICHLORODIFLUOROMETHANE	75-71-8	10.00	70.00	130.00		20.00
DIETHYL ETHER	60-29-7	10.00	70.00	130.00		20.00

Analyte Name	Analyte Label (CAS)	Lower Rejection (%)	Lower Recovery (%)	Upper Recovery (%)	Upper Rejection (%)	RPD (%)
Method: 524.2	**Matrix:** Water					
QC Type: Laboratory_Control_Sample						
DI-ISOPROPYL ETHER	108-20-3	10.00	70.00	130.00		20.00
ETHYLBENZENE	100-41-4	10.00	70.00	130.00		20.00
HEXACHLOROBUTADIENE	87-68-3	10.00	70.00	130.00		20.00
ISOPROPYLBENZENE	98-82-8	10.00	70.00	130.00		20.00
M,P-XYLENES	1330-20-7	10.00	70.00	130.00		20.00
METHYL TERT-BUTYL ETHER	1634-04-4	10.00	70.00	130.00		20.00
METHYLENE CHLORIDE	75-09-2	10.00	70.00	130.00		20.00
NAPHTHALENE	91-20-3	10.00	70.00	130.00		20.00
N-BUTYLBENZENE	104-51-8	10.00	70.00	130.00		20.00
N-PROPYLBENZENE	103-65-1	10.00	70.00	130.00		20.00
O-XYLENE	95-47-6	10.00	70.00	130.00		20.00
P-ISOPROPYLTOLUENE	99-87-6	10.00	70.00	130.00		20.00
SEC-BUTYLBENZENE	135-98-8	10.00	70.00	130.00		20.00
STYRENE	100-42-5	10.00	70.00	130.00		20.00
TERT-BUTYLBENZENE	98-06-6	10.00	70.00	130.00		20.00
TETRACHLOROETHENE	127-18-4	10.00	70.00	130.00		20.00
TOLUENE	108-88-3	10.00	70.00	130.00		20.00
TRANS-1,2-DICHLOROETHENE	156-60-5	10.00	70.00	130.00		20.00
TRANS-1,3-DICHLOROPROPENE	10061-02-6	10.00	70.00	130.00		20.00
TRICHLOROETHENE	79-01-6	10.00	70.00	130.00		20.00
TRICHLOROFLUOROMETHANE	75-69-4	10.00	70.00	130.00		20.00
VINYL ACETATE	108-05-4	10.00	70.00	130.00		20.00
VINYL CHLORIDE	75-01-4	10.00	70.00	130.00		20.00
QC Type: Matrix_Spike						
1,1,1,2-TETRACHLOROETHANE	630-20-6	10.00	70.00	130.00		20.00
1,1,1-TRICHLOROETHANE	71-55-6	10.00	70.00	130.00		20.00
1,1,2,2-TETRACHLOROETHANE	79-34-5	10.00	70.00	130.00		20.00
1,1,2-TRICHLOROETHANE	79-00-5	10.00	70.00	130.00		20.00
1,1-DICHLOROETHANE	75-34-3	10.00	70.00	130.00		20.00
1,1-DICHLOROETHENE	75-35-4	10.00	70.00	130.00		20.00
1,1-DICHLOROPROPENE	563-58-6	10.00	70.00	130.00		20.00

Analyte Name	Analyte Label (CAS)	Lower Rejection (%)	Lower Recovery (%)	Upper Recovery (%)	Upper Rejection (%)	RPD (%)
Method: 524.2	**Matrix: Water**					
QC Type: Matrix_Spike						
1,2,3-TRICHLOROBENZENE	87-61-6	10.00	70.00	130.00		20.00
1,2,3-TRICHLOROPROPANE	96-18-4	10.00	70.00	130.00		20.00
1,2,4-TRICHLOROBENZENE	120-82-1	10.00	70.00	130.00		20.00
1,2,4-TRIMETHYLBENZENE	95-63-6	10.00	70.00	130.00		20.00
1,2-DIBROMO-3-CHLOROPROPANE	96-12-8	10.00	70.00	130.00		20.00
1,2-DIBROMOETHANE	106-93-4	10.00	70.00	130.00		20.00
1,2-DICHLOROBENZENE	95-50-1	10.00	70.00	130.00		20.00
1,2-DICHLOROETHANE	107-06-2	10.00	70.00	130.00		20.00
1,2-DICHLOROPROPANE	78-87-5	10.00	70.00	130.00		20.00
1,3,5-TRIMETHYLBENZENE	108-67-8	10.00	70.00	130.00		20.00
1,3-DICHLOROBENZENE	541-73-1	10.00	70.00	130.00		20.00
1,3-DICHLOROPROPANE	142-28-9	10.00	70.00	130.00		20.00
1,4-DICHLOROBENZENE	106-46-7	10.00	70.00	130.00		20.00
1,4-DIOXANE	123-91-1	10.00	70.00	130.00		20.00
2,2-DICHLOROPROPANE	594-20-7	10.00	70.00	130.00		20.00
2-BUTANONE	78-93-3	10.00	70.00	130.00		20.00
2-CHLOROTOLUENE	95-49-8	10.00	70.00	130.00		20.00
2-HEXANONE	591-78-6	10.00	70.00	130.00		20.00
4-CHLOROTOLUENE	106-43-4	10.00	70.00	130.00		20.00
4-METHYL-2-PENTANONE	108-10-1	10.00	70.00	130.00		20.00
ACETONE	67-64-1	10.00	70.00	130.00		20.00
BENZENE	71-43-2	10.00	70.00	130.00		20.00
BROMOBENZENE	108-86-1	10.00	70.00	130.00		20.00
BROMOCHLOROMETHANE	74-97-5	10.00	70.00	130.00		20.00
BROMODICHLOROMETHANE	75-27-4	10.00	70.00	130.00		20.00
BROMOFORM	75-25-2	10.00	70.00	130.00		20.00
BROMOMETHANE	74-83-9	10.00	70.00	130.00		20.00
CARBON DISULFIDE	75-15-0	10.00	70.00	130.00		20.00
CARBON TETRACHLORIDE	56-23-5	10.00	70.00	130.00		20.00
CHLOROBENZENE	108-90-7	10.00	70.00	130.00		20.00
CHLOROETHANE	75-00-3	10.00	70.00	130.00		20.00
CHLOROFORM	67-66-3	10.00	70.00	130.00		20.00

Analyte Name	Analyte Label (CAS)	Lower Rejection (%)	Lower Recovery (%)	Upper Recovery (%)	Upper Rejection (%)	RPD (%)
Method: 524.2	**Matrix: Water**					
QC Type: Matrix_Spike						
CHLOROMETHANE	74-87-3	10.00	70.00	130.00		20.00
CIS-1,2-DICHLOROETHENE	156-59-2	10.00	70.00	130.00		20.00
CIS-1,3-DICHLOROPROPENE	10061-01-5	10.00	70.00	130.00		20.00
DIBROMOCHLOROMETHANE	124-48-1	10.00	70.00	130.00		20.00
DIBROMOMETHANE	74-95-3	10.00	70.00	130.00		20.00
DICHLORODIFLUOROMETHANE	75-71-8	10.00	70.00	130.00		20.00
DIETHYL ETHER	60-29-7	10.00	70.00	130.00		20.00
DI-ISOPROPYL ETHER	108-20-3	10.00	70.00	130.00		20.00
ETHYLBENZENE	100-41-4	10.00	70.00	130.00		20.00
HEXACHLOROBUTADIENE	87-68-3	10.00	70.00	130.00		20.00
ISOPROPYLBENZENE	98-82-8	10.00	70.00	130.00		20.00
M,P-XYLENES	1330-20-7	10.00	70.00	130.00		20.00
METHYL TERT-BUTYL ETHER	1634-04-4	10.00	70.00	130.00		20.00
METHYLENE CHLORIDE	75-09-2	10.00	70.00	130.00		20.00
NAPHTHALENE	91-20-3	10.00	70.00	130.00		20.00
N-BUTYLBENZENE	104-51-8	10.00	70.00	130.00		20.00
N-PROPYLBENZENE	103-65-1	10.00	70.00	130.00		20.00
O-XYLENE	95-47-6	10.00	70.00	130.00		20.00
P-ISOPROPYLTOLUENE	99-87-6	10.00	70.00	130.00		20.00
SEC-BUTYLBENZENE	135-98-8	10.00	70.00	130.00		20.00
STYRENE	100-42-5	10.00	70.00	130.00		20.00
TERT-BUTYLBENZENE	98-06-6	10.00	70.00	130.00		20.00
TETRACHLOROETHENE	127-18-4	10.00	70.00	130.00		20.00
TOLUENE	108-88-3	10.00	70.00	130.00		20.00
TRANS-1,2-DICHLOROETHENE	156-60-5	10.00	70.00	130.00		20.00
TRANS-1,3-DICHLOROPROPENE	10061-02-6	10.00	70.00	130.00		20.00
TRICHLOROETHENE	79-01-6	10.00	70.00	130.00		20.00
TRICHLOROFLUOROMETHANE	75-69-4	10.00	70.00	130.00		20.00
VINYL ACETATE	108-05-4	10.00	70.00	130.00		20.00
VINYL CHLORIDE	75-01-4	10.00	70.00	130.00		20.00

Analyte Name	Analyte Label (CAS)	Lower Rejection (%)	Lower Recovery (%)	Upper Recovery (%)	Upper Rejection (%)	RPD (%)
Method: 524.2	**Matrix: Water**					
QC Type: Surrogate						
1,2-DICHLOROBENZENE-D4	2199-69-1	10.00	80	120		
4-Bromofluorobenzene	460-00-4	10.00	80	130		
Method: 6010B	**Matrix: Water**					
QC Type: Laboratory_Control_Sample						
ALUMINUM	7429-90-5	50.00	80.00	120.00		20.00
BARIUM	7440-39-3	50.00	80.00	120.00		20.00
BERYLLIUM	7440-41-7	50.00	80.00	120.00		20.00
CADMIUM	7440-43-9	50.00	80.00	120.00		20.00
CALCIUM	7440-70-2	50.00	80.00	120.00		20.00
CHROMIUM	7440-47-3	50.00	80.00	120.00		20.00
COBALT	7440-48-4	50.00	80.00	120.00		20.00
COPPER	7440-50-8	50.00	80.00	120.00		20.00
IRON	7439-89-6	50.00	80.00	120.00		20.00
LEAD	7439-92-1	50.00	80.00	120.00		20.00
MAGNESIUM	7439-95-4	50.00	80.00	120.00		20.00
MANGANESE	7439-96-5	50.00	80.00	120.00		20.00
NICKEL	7440-02-0	50.00	80.00	120.00		20.00
POTASSIUM	7440-09-7	50.00	80.00	120.00		20.00
SELENIUM	7782-49-2	50.00	80.00	120.00		20.00
SILVER	7440-22-4	50.00	80.00	120.00		20.00
VANADIUM	7440-62-2	50.00	80.00	120.00		20.00
ZINC	7440-66-6	50.00	80.00	120.00		20.00
QC Type: Matrix_Spike						
ALUMINUM	7429-90-5	50.00	80.00	120.00		20.00
BARIUM	7440-39-3	50.00	80.00	120.00		20.00
BERYLLIUM	7440-41-7	50.00	80.00	120.00		20.00
CADMIUM	7440-43-9	50.00	80.00	120.00		20.00
CALCIUM	7440-70-2	50.00	80.00	120.00		20.00
CHROMIUM	7440-47-3	50.00	80.00	120.00		20.00
COBALT	7440-48-4	50.00	80.00	120.00		20.00

Analyte Name	Analyte Label (CAS)	Lower Rejection (%)	Lower Recovery (%)	Upper Recovery (%)	Upper Rejection (%)	RPD (%)
Method: 6010B **Matrix: Water**						
QC Type: Matrix_Spike						
COPPER	7440-50-8	50.00	80.00	120.00		20.00
IRON	7439-89-6	50.00	80.00	120.00		20.00
LEAD	7439-92-1	50.00	80.00	120.00		20.00
MAGNESIUM	7439-95-4	50.00	80.00	120.00		20.00
MANGANESE	7439-96-5	50.00	80.00	120.00		20.00
NICKEL	7440-02-0	50.00	80.00	120.00		20.00
POTASSIUM	7440-09-7	50.00	80.00	120.00		20.00
SELENIUM	7782-49-2	50.00	80.00	120.00		20.00
SILVER	7440-22-4	50.00	80.00	120.00		20.00
VANADIUM	7440-62-2	50.00	80.00	120.00		20.00
ZINC	7440-66-6	50.00	80.00	120.00		20.00
Method: 6010B-Ca **Matrix: Water**						
QC Type: Laboratory_Control_Sample						
HARDNESS (AS CACO3)	HARD	50.00	80.00	120.00		20.00
QC Type: Matrix_Spike						
HARDNESS (AS CACO3)	HARD	30.00	80.00	120.00		20.00
Method: 7041 **Matrix: Water**						
QC Type: Laboratory_Control_Sample						
ANTIMONY	7440-36-0	50.00	80.00	120.00		20.00
QC Type: Matrix_Spike						
ANTIMONY	7440-36-0	50.00	80.00	120.00		20.00
Method: 7060A **Matrix: Water**						
QC Type: Laboratory_Control_Sample						
ARSENIC	7440-38-2	50.00	80.00	120.00		20.00

Analyte Name	Analyte Label (CAS)	Lower Rejection (%)	Lower Recovery (%)	Upper Recovery (%)	Upper Rejection (%)	RPD (%)
Method: 7060A **Matrix: Water**						
QC Type: Matrix_Spike						
ARSENIC	7440-38-2	50.00	80.00	120.00		20.00
Method: 7470A **Matrix: Water**						
QC Type: Laboratory_Control_Sample						
MERCURY	7439-97-6	50.00	80.00	120.00		20.00
QC Type: Matrix_Spike						
MERCURY	7439-97-6	50.00	80.00	120.00		20.00
Method: 7841 **Matrix: Water**						
QC Type: Laboratory_Control_Sample						
THALLIUM	7440-28-0	50.00	80.00	120.00		20.00
QC Type: Matrix_Spike						
THALLIUM	7440-28-0	50.00	80.00	120.00		20.00
Method: 8100M **Matrix: Soil**						
QC Type: Laboratory_Control_Sample						
PETROLEUM HYDROCARBONS (TPH)	PHC	10.00	40.00	140.00		50.00
QC Type: Matrix_Spike						
PETROLEUM HYDROCARBONS (TPH)	PHC	10.00	40.00	140.00		50.00
QC Type: Surrogate						
O-Terphenyl	84-15-1	10.00	40	140		
Method: 8260B **Matrix: Soil**						
QC Type: Laboratory_Control_Sample						
1,1,1,2-TETRACHLOROETHANE	630-20-6	10.00	75.00	125.00		30.00

Analyte Name	Analyte Label (CAS)	Lower Rejection (%)	Lower Recovery (%)	Upper Recovery (%)	Upper Rejection (%)	RPD (%)
Method: 8260B **Matrix:** Soil						
QC Type: Laboratory_Control_Sample						
1,1,1-TRICHLOROETHANE	71-55-6	10.00	70.00	135.00		30.00
1,1,2,2-TETRACHLOROETHANE	79-34-5	10.00	55.00	130.00		30.00
1,1,2-TRICHLOROETHANE	79-00-5	10.00	60.00	125.00		30.00
1,1-DICHLOROETHANE	75-34-3	10.00	75.00	125.00		30.00
1,1-DICHLOROETHENE	75-35-4	10.00	65.00	135.00		30.00
1,1-DICHLOROPROPENE	563-58-6	10.00	70.00	135.00		30.00
1,2,3-TRICHLOROBENZENE	87-61-6	10.00	60.00	135.00		30.00
1,2,3-TRICHLOROPROPANE	96-18-4	10.00	65.00	130.00		30.00
1,2,4-TRICHLOROBENZENE	120-82-1	10.00	65.00	130.00		30.00
1,2,4-TRIMETHYLBENZENE	95-63-6	10.00	65.00	135.00		30.00
1,2-DIBROMO-3-CHLOROPROPANE	96-12-8	10.00	40.00	135.00		30.00
1,2-DIBROMOETHANE	106-93-4	10.00	70.00	125.00		30.00
1,2-DICHLOROBENZENE	95-50-1	10.00	75.00	120.00		30.00
1,2-DICHLOROETHANE	107-06-2	10.00	70.00	135.00		30.00
1,2-DICHLOROPROPANE	78-87-5	10.00	70.00	120.00		30.00
1,3,5-TRIMETHYLBENZENE	108-67-8	10.00	65.00	135.00		30.00
1,3-DICHLOROBENZENE	541-73-1	10.00	70.00	125.00		30.00
1,3-DICHLOROPROPANE	142-28-9	10.00	75.00	125.00		30.00
1,4-DICHLOROBENZENE	106-46-7	10.00	70.00	125.00		30.00
1,4-DIOXANE	123-91-1	10.00	70.00	130.00		20.00
2,2-DICHLOROPROPANE	594-20-7	10.00	65.00	135.00		30.00
2-BUTANONE	78-93-3	10.00	30.00	160.00		30.00
2-CHLOROTOLUENE	95-49-8	10.00	70.00	130.00		30.00
2-HEXANONE	591-78-6	10.00	45.00	145.00		30.00
4-CHLOROTOLUENE	106-43-4	10.00	75.00	125.00		30.00
4-ISOPROPYLTOLUENE	99-87-6	10.00	75.00	135.00		30.00
4-METHYL-2-PENTANONE	108-10-1	10.00	45.00	145.00		30.00
ACETONE	67-64-1	10.00	20.00	160.00		30.00
BENZENE	71-43-2	10.00	75.00	125.00		30.00
BROMOBENZENE	108-86-1	10.00	65.00	120.00		30.00
BROMOCHLOROMETHANE	74-97-5	10.00	70.00	125.00		30.00
BROMODICHLOROMETHANE	75-27-4	10.00	70.00	130.00		30.00

Analyte Name	Analyte Label (CAS)	Lower Rejection (%)	Lower Recovery (%)	Upper Recovery (%)	Upper Rejection (%)	RPD (%)
Method: 8260B	**Matrix:** Soil					
QC Type: Laboratory_Control_Sample						
BROMOFORM	75-25-2	10.00	55.00	135.00		30.00
BROMOMETHANE	74-83-9	10.00	30.00	160.00		30.00
CARBON DISULFIDE	75-15-0	10.00	45.00	160.00		30.00
CARBON TETRACHLORIDE	56-23-5	10.00	65.00	135.00		30.00
CHLOROBENZENE	108-90-7	10.00	75.00	125.00		30.00
CHLOROETHANE	75-00-3	10.00	40.00	155.00		30.00
CHLOROFORM	67-66-3	10.00	70.00	125.00		30.00
CHLOROMETHANE	74-87-3	10.00	50.00	130.00		30.00
CIS-1,2-DICHLOROETHENE	156-59-2	10.00	65.00	125.00		30.00
CIS-1,3-DICHLOROPROPENE	10061-01-5	10.00	70.00	125.00		30.00
DIBROMOCHLOROMETHANE	124-48-1	10.00	65.00	130.00		30.00
DIBROMOMETHANE	74-95-3	10.00	75.00	130.00		30.00
DICHLORODIFLUOROMETHANE	75-71-8	10.00	35.00	135.00		30.00
DIETHYL ETHER	60-29-7	10.00	70.00	130.00		30.00
DI-ISOPROPYL ETHER	108-20-3	10.00	70.00	130.00		30.00
ETHYLBENZENE	100-41-4	10.00	75.00	125.00		30.00
HEXACHLOROBUTADIENE	87-68-3	10.00	55.00	140.00		30.00
ISOPROPYLBENZENE	98-82-8	10.00	75.00	130.00		30.00
M,P-XYLENES	1330-20-7	10.00	80.00	125.00		30.00
METHYL TERT-BUTYL ETHER	1634-04-4	10.00	70.00	130.00		30.00
METHYLENE CHLORIDE	75-09-2	10.00	55.00	140.00		30.00
NAPHTHALENE	91-20-3	10.00	40.00	125.00		30.00
N-BUTYLBENZENE	104-51-8	10.00	65.00	140.00		30.00
N-PROPYLBENZENE	103-65-1	10.00	65.00	135.00		30.00
O-XYLENE	95-47-6	10.00	75.00	125.00		30.00
SEC-BUTYLBENZENE	135-98-8	10.00	65.00	130.00		30.00
STYRENE	100-42-5	10.00	75.00	125.00		30.00
TERT-BUTYLBENZENE	98-06-6	10.00	65.00	130.00		30.00
TETRACHLOROETHENE	127-18-4	10.00	65.00	140.00		30.00
TOLUENE	108-88-3	10.00	70.00	125.00		30.00
TRANS-1,2-DICHLOROETHENE	156-60-5	10.00	65.00	135.00		30.00
TRANS-1,3-DICHLOROPROPENE	10061-02-6	10.00	65.00	125.00		30.00

Analyte Name	Analyte Label (CAS)	Lower Rejection (%)	Lower Recovery (%)	Upper Recovery (%)	Upper Rejection (%)	RPD (%)
Method: 8260B	**Matrix: Soil**					
QC Type: Laboratory_Control_Sample						
TRICHLOROETHENE	79-01-6	10.00	75.00	125.00		30.00
TRICHLOROFLUOROMETHANE	75-69-4	10.00	25.00	185.00		30.00
VINYL ACETATE	108-05-4	10.00	70.00	130.00		20.00
VINYL CHLORIDE	75-01-4	10.00	60.00	125.00		30.00
QC Type: Matrix_Spike						
1,1,1,2-TETRACHLOROETHANE	630-20-6	10.00	75.00	125.00		30.00
1,1,1-TRICHLOROETHANE	71-55-6	10.00	70.00	135.00		30.00
1,1,2,2-TETRACHLOROETHANE	79-34-5	10.00	55.00	130.00		30.00
1,1,2-TRICHLOROETHANE	79-00-5	10.00	60.00	125.00		30.00
1,1-DICHLOROETHANE	75-34-3	10.00	75.00	125.00		30.00
1,1-DICHLOROETHENE	75-35-4	10.00	65.00	135.00		30.00
1,1-DICHLOROPROPENE	563-58-6	10.00	70.00	135.00		30.00
1,2,3-TRICHLOROBENZENE	87-61-6	10.00	60.00	135.00		30.00
1,2,3-TRICHLOROPROPANE	96-18-4	10.00	65.00	130.00		30.00
1,2,4-TRICHLOROBENZENE	120-82-1	10.00	65.00	130.00		30.00
1,2,4-TRIMETHYLBENZENE	95-63-6	10.00	65.00	135.00		30.00
1,2-DIBROMO-3-CHLOROPROPANE	96-12-8	10.00	40.00	135.00		30.00
1,2-DIBROMOETHANE	106-93-4	10.00	70.00	125.00		30.00
1,2-DICHLOROBENZENE	95-50-1	10.00	75.00	120.00		30.00
1,2-DICHLOROETHANE	107-06-2	10.00	70.00	135.00		30.00
1,2-DICHLOROPROPANE	78-87-5	10.00	70.00	120.00		30.00
1,3,5-TRIMETHYLBENZENE	108-67-8	10.00	65.00	135.00		30.00
1,3-DICHLOROBENZENE	541-73-1	10.00	70.00	125.00		30.00
1,3-DICHLOROPROPANE	142-28-9	10.00	75.00	125.00		30.00
1,4-DICHLOROBENZENE	106-46-7	10.00	70.00	125.00		30.00
1,4-DIOXANE	123-91-1	10.00	70.00	130.00		20.00
2,2-DICHLOROPROPANE	594-20-7	10.00	65.00	135.00		30.00
2-BUTANONE	78-93-3	10.00	30.00	160.00		30.00
2-CHLOROTOLUENE	95-49-8	10.00	70.00	130.00		30.00
2-HEXANONE	591-78-6	10.00	45.00	145.00		30.00
4-CHLOROTOLUENE	106-43-4	10.00	75.00	125.00		30.00

Analyte Name	Analyte Label (CAS)	Lower Rejection (%)	Lower Recovery (%)	Upper Recovery (%)	Upper Rejection (%)	RPD (%)
Method: 8260B	**Matrix:** Soil					
QC Type: Matrix_Spike						
4-ISOPROPYLTOLUENE	99-87-6	10.00	75.00	135.00		30.00
4-METHYL-2-PENTANONE	108-10-1	10.00	45.00	145.00		30.00
ACETONE	67-64-1	10.00	20.00	160.00		30.00
BENZENE	71-43-2	10.00	75.00	125.00		30.00
BROMOBENZENE	108-86-1	10.00	65.00	120.00		30.00
BROMOCHLOROMETHANE	74-97-5	10.00	70.00	125.00		30.00
BROMODICHLOROMETHANE	75-27-4	10.00	70.00	130.00		30.00
BROMOFORM	75-25-2	10.00	55.00	135.00		30.00
BROMOMETHANE	74-83-9	10.00	30.00	160.00		30.00
CARBON DISULFIDE	75-15-0	10.00	45.00	160.00		30.00
CARBON TETRACHLORIDE	56-23-5	10.00	65.00	135.00		30.00
CHLOROBENZENE	108-90-7	10.00	75.00	125.00		30.00
CHLOROETHANE	75-00-3	10.00	40.00	155.00		30.00
CHLOROFORM	67-66-3	10.00	70.00	125.00		30.00
CHLOROMETHANE	74-87-3	10.00	50.00	130.00		30.00
CIS-1,2-DICHLOROETHENE	156-59-2	10.00	65.00	125.00		30.00
CIS-1,3-DICHLOROPROPENE	10061-01-5	10.00	70.00	125.00		30.00
DIBROMOCHLOROMETHANE	124-48-1	10.00	65.00	130.00		30.00
DIBROMOMETHANE	74-95-3	10.00	75.00	130.00		30.00
DICHLORODIFLUOROMETHANE	75-71-8	10.00	35.00	135.00		30.00
DIETHYL ETHER	60-29-7	10.00	70.00	130.00		30.00
DI-ISOPROPYL ETHER	108-20-3	10.00	70.00	130.00		30.00
ETHYLBENZENE	100-41-4	10.00	75.00	125.00		30.00
HEXACHLOROBUTADIENE	87-68-3	10.00	55.00	140.00		30.00
ISOPROPYLBENZENE	98-82-8	10.00	75.00	130.00		30.00
M,P-XYLENES	1330-20-7	10.00	80.00	125.00		30.00
METHYL TERT-BUTYL ETHER	1634-04-4	10.00	70.00	130.00		30.00
METHYLENE CHLORIDE	75-09-2	10.00	55.00	140.00		30.00
NAPHTHALENE	91-20-3	10.00	40.00	125.00		30.00
N-BUTYLBENZENE	104-51-8	10.00	65.00	140.00		30.00
N-PROPYLBENZENE	103-65-1	10.00	65.00	135.00		30.00
O-XYLENE	95-47-6	10.00	75.00	125.00		30.00

Analyte Name	Analyte Label (CAS)	Lower Rejection (%)	Lower Recovery (%)	Upper Recovery (%)	Upper Rejection (%)	RPD (%)
Method: 8260B	**Matrix: Soil**					
QC Type: Matrix_Spike						
SEC-BUTYLBENZENE	135-98-8	10.00	65.00	130.00		30.00
STYRENE	100-42-5	10.00	75.00	125.00		30.00
TERT-BUTYLBENZENE	98-06-6	10.00	65.00	130.00		30.00
TETRACHLOROETHENE	127-18-4	10.00	65.00	140.00		30.00
TOLUENE	108-88-3	10.00	70.00	125.00		30.00
TRANS-1,2-DICHLOROETHENE	156-60-5	10.00	65.00	135.00		30.00
TRANS-1,3-DICHLOROPROPENE	10061-02-6	10.00	65.00	125.00		30.00
TRICHLOROETHENE	79-01-6	10.00	75.00	125.00		30.00
TRICHLOROFLUOROMETHANE	75-69-4	10.00	25.00	185.00		30.00
VINYL ACETATE	108-05-4	10.00	70.00	130.00		20.00
VINYL CHLORIDE	75-01-4	10.00	60.00	125.00		30.00
QC Type: Surrogate						
1,2-DICHLOROETHANE-D4	17060-07-0	10.00	70	120		
4-BROMOFLUOROBENZENE (SURR)	460-00-4	10.00	85	115		
DIBROMOFLUOROMETHANE	1868-53-7	10.00	85	115		
TOLUENE-D8	2037-26-5	10.00	85	115		
Method: 8260B	**Matrix: Water**					
QC Type: Laboratory_Control_Sample						
1,1,1,2-TETRACHLOROETHANE	630-20-6	10.00	80.00	130.00		30.00
1,1,1-TRICHLOROETHANE	71-55-6	10.00	65.00	130.00		30.00
1,1,2,2-TETRACHLOROETHANE	79-34-5	10.00	65.00	130.00		30.00
1,1,2-TRICHLOROETHANE	79-00-5	10.00	75.00	125.00		30.00
1,1-DICHLOROETHANE	75-34-3	10.00	70.00	135.00		30.00
1,1-DICHLOROETHENE	75-35-4	10.00	70.00	130.00		30.00
1,1-DICHLOROPROPENE	563-58-6	10.00	75.00	130.00		30.00
1,2,3-TRICHLOROBENZENE	87-61-6	10.00	55.00	140.00		30.00
1,2,3-TRICHLOROPROPANE	96-18-4	10.00	75.00	125.00		30.00
1,2,4-TRICHLOROBENZENE	120-82-1	10.00	65.00	135.00		30.00
1,2,4-TRIMETHYLBENZENE	95-63-6	10.00	75.00	130.00		30.00
1,2-DIBROMO-3-CHLOROPROPANE	96-12-8	10.00	50.00	130.00		30.00

Analyte Name	Analyte Label (CAS)	Lower Rejection (%)	Lower Recovery (%)	Upper Recovery (%)	Upper Rejection (%)	RPD (%)
Method: 8260B	**Matrix: Water**					
QC Type: Laboratory_Control_Sample						
1,2-DIBROMOETHANE	106-93-4	10.00	80.00	120.00		30.00
1,2-DICHLOROBENZENE	95-50-1	10.00	70.00	120.00		30.00
1,2-DICHLOROETHANE	107-06-2	10.00	70.00	130.00		30.00
1,2-DICHLOROPROPANE	78-87-5	10.00	75.00	125.00		30.00
1,3,5-TRIMETHYLBENZENE	108-67-8	10.00	75.00	130.00		30.00
1,3-DICHLOROBENZENE	541-73-1	10.00	75.00	125.00		30.00
1,3-DICHLOROPROPANE	142-28-9	10.00	75.00	125.00		30.00
1,4-DICHLOROBENZENE	106-46-7	10.00	75.00	125.00		30.00
1,4-DIOXANE	123-91-1	10.00	0.00	200.00		30.00
2,2-DICHLOROPROPANE	594-20-7	10.00	70.00	135.00		30.00
2-BUTANONE	78-93-3	10.00	30.00	150.00		30.00
2-CHLOROTOLUENE	95-49-8	10.00	75.00	125.00		30.00
2-HEXANONE	591-78-6	10.00	55.00	130.00		30.00
4-CHLOROTOLUENE	106-43-4	10.00	75.00	130.00		30.00
4-ISOPROPYLTOLUENE	99-87-6	10.00	75.00	130.00		30.00
4-METHYL-2-PENTANONE	108-10-1	10.00	60.00	135.00		30.00
ACETONE	67-64-1	10.00	40.00	140.00		30.00
BENZENE	71-43-2	10.00	80.00	120.00		30.00
BROMOBENZENE	108-86-1	10.00	75.00	125.00		30.00
BROMOCHLOROMETHANE	74-97-5	10.00	65.00	130.00		30.00
BROMODICHLOROMETHANE	75-27-4	10.00	75.00	120.00		30.00
BROMOFORM	75-25-2	10.00	70.00	130.00		30.00
BROMOMETHANE	74-83-9	10.00	30.00	145.00		30.00
CARBON DISULFIDE	75-15-0	10.00	35.00	160.00		30.00
CARBON TETRACHLORIDE	56-23-5	10.00	65.00	140.00		30.00
CHLOROBENZENE	108-90-7	10.00	80.00	120.00		30.00
CHLOROETHANE	75-00-3	10.00	60.00	135.00		30.00
CHLOROFORM	67-66-3	10.00	65.00	135.00		30.00
CHLOROMETHANE	74-87-3	10.00	40.00	125.00		30.00
CIS-1,2-DICHLOROETHENE	156-59-2	10.00	70.00	125.00		30.00
CIS-1,3-DICHLOROPROPENE	10061-01-5	10.00	70.00	130.00		30.00
DIBROMOCHLOROMETHANE	124-48-1	10.00	60.00	135.00		30.00

Analyte Name	Analyte Label (CAS)	Lower Rejection (%)	Lower Recovery (%)	Upper Recovery (%)	Upper Rejection (%)	RPD (%)
Method: 8260B	**Matrix:** Water					
QC Type: Laboratory_Control_Sample						
DIBROMOMETHANE	74-95-3	10.00	75.00	125.00		30.00
DICHLORODIFLUOROMETHANE	75-71-8	10.00	30.00	155.00		30.00
DIETHYL ETHER	60-29-7	10.00	70.00	130.00		30.00
DI-ISOPROPYL ETHER	108-20-3	10.00	70.00	130.00		30.00
ETHYLBENZENE	100-41-4	10.00	75.00	125.00		30.00
HEXACHLOROBUTADIENE	87-68-3	10.00	50.00	140.00		30.00
ISOPROPYLBENZENE	98-82-8	10.00	75.00	125.00		30.00
M,P-XYLENES	1330-20-7	10.00	75.00	130.00		30.00
METHYL TERT-BUTYL ETHER	1634-04-4	10.00	65.00	125.00		30.00
METHYLENE CHLORIDE	75-09-2	10.00	55.00	140.00		30.00
NAPHTHALENE	91-20-3	10.00	55.00	140.00		30.00
N-BUTYLBENZENE	104-51-8	10.00	70.00	135.00		30.00
N-PROPYLBENZENE	103-65-1	10.00	70.00	130.00		30.00
O-XYLENE	95-47-6	10.00	80.00	120.00		30.00
SEC-BUTYLBENZENE	135-98-8	10.00	70.00	125.00		30.00
STYRENE	100-42-5	10.00	65.00	135.00		30.00
TERT-BUTYLBENZENE	98-06-6	10.00	70.00	130.00		30.00
TETRACHLOROETHENE	127-18-4	10.00	45.00	150.00		30.00
TOLUENE	108-88-3	10.00	75.00	120.00		30.00
TRANS-1,2-DICHLOROETHENE	156-60-5	10.00	60.00	140.00		30.00
TRANS-1,3-DICHLOROPROPENE	10061-02-6	10.00	55.00	140.00		30.00
TRICHLOROETHENE	79-01-6	10.00	70.00	125.00		30.00
TRICHLOROFLUOROMETHANE	75-69-4	10.00	60.00	145.00		30.00
VINYL ACETATE	108-05-4	10.00	70.00	130.00		30.00
VINYL CHLORIDE	75-01-4	10.00	50.00	145.00		30.00
QC Type: Matrix_Spike						
1,1,1,2-TETRACHLOROETHANE	630-20-6	10.00	80.00	130.00		30.00
1,1,1-TRICHLOROETHANE	71-55-6	10.00	65.00	130.00		30.00
1,1,2,2-TETRACHLOROETHANE	79-34-5	10.00	65.00	130.00		30.00
1,1,2-TRICHLOROETHANE	79-00-5	10.00	75.00	125.00		30.00
1,1-DICHLOROETHANE	75-34-3	10.00	70.00	135.00		30.00

Analyte Name	Analyte Label (CAS)	Lower Rejection (%)	Lower Recovery (%)	Upper Recovery (%)	Upper Rejection (%)	RPD (%)
Method: 8260B	**Matrix:** Water					
QC Type: Matrix_Spike						
1,1-DICHLOROETHENE	75-35-4	10.00	70.00	130.00		30.00
1,1-DICHLOROPROPENE	563-58-6	10.00	75.00	130.00		30.00
1,2,3-TRICHLOROBENZENE	87-61-6	10.00	55.00	140.00		30.00
1,2,3-TRICHLOROPROPANE	96-18-4	10.00	75.00	125.00		30.00
1,2,4-TRICHLOROBENZENE	120-82-1	10.00	65.00	135.00		30.00
1,2,4-TRIMETHYLBENZENE	95-63-6	10.00	75.00	130.00		30.00
1,2-DIBROMO-3-CHLOROPROPANE	96-12-8	10.00	50.00	130.00		30.00
1,2-DIBROMOETHANE	106-93-4	10.00	80.00	120.00		30.00
1,2-DICHLOROBENZENE	95-50-1	10.00	70.00	120.00		30.00
1,2-DICHLOROETHANE	107-06-2	10.00	70.00	130.00		30.00
1,2-DICHLOROPROPANE	78-87-5	10.00	75.00	125.00		30.00
1,3,5-TRIMETHYLBENZENE	108-67-8	10.00	75.00	130.00		30.00
1,3-DICHLOROBENZENE	541-73-1	10.00	75.00	125.00		30.00
1,3-DICHLOROPROPANE	142-28-9	10.00	75.00	125.00		30.00
1,4-DICHLOROBENZENE	106-46-7	10.00	75.00	125.00		30.00
1,4-DIOXANE	123-91-1	10.00	70.00	130.00		30.00
2,2-DICHLOROPROPANE	594-20-7	10.00	70.00	135.00		30.00
2-BUTANONE	78-93-3	10.00	30.00	150.00		30.00
2-CHLOROTOLUENE	95-49-8	10.00	75.00	125.00		30.00
2-HEXANONE	591-78-6	10.00	55.00	130.00		30.00
4-CHLOROTOLUENE	106-43-4	10.00	75.00	130.00		30.00
4-ISOPROPYLTOLUENE	99-87-6	10.00	75.00	130.00		30.00
4-METHYL-2-PENTANONE	108-10-1	10.00	60.00	135.00		30.00
ACETONE	67-64-1	10.00	40.00	140.00		30.00
BENZENE	71-43-2	10.00	80.00	120.00		30.00
BROMOBENZENE	108-86-1	10.00	75.00	125.00		30.00
BROMOCHLOROMETHANE	74-97-5	10.00	65.00	130.00		30.00
BROMODICHLOROMETHANE	75-27-4	10.00	75.00	120.00		30.00
BROMOFORM	75-25-2	10.00	70.00	130.00		30.00
BROMOMETHANE	74-83-9	10.00	30.00	145.00		30.00
CARBON DISULFIDE	75-15-0	10.00	35.00	160.00		30.00
CARBON TETRACHLORIDE	56-23-5	10.00	65.00	140.00		30.00

Analyte Name	Analyte Label (CAS)	Lower Rejection (%)	Lower Recovery (%)	Upper Recovery (%)	Upper Rejection (%)	RPD (%)
Method: 8260B	**Matrix:** Water					
QC Type: Matrix_Spike						
CHLOROBENZENE	108-90-7	10.00	80.00	120.00		30.00
CHLOROETHANE	75-00-3	10.00	60.00	135.00		30.00
CHLOROFORM	67-66-3	10.00	65.00	135.00		30.00
CHLOROMETHANE	74-87-3	10.00	40.00	125.00		30.00
CIS-1,2-DICHLOROETHENE	156-59-2	10.00	70.00	125.00		30.00
CIS-1,3-DICHLOROPROPENE	10061-01-5	10.00	70.00	130.00		30.00
DIBROMOCHLOROMETHANE	124-48-1	10.00	60.00	135.00		30.00
DIBROMOMETHANE	74-95-3	10.00	75.00	125.00		30.00
DICHLORODIFLUOROMETHANE	75-71-8	10.00	30.00	155.00		30.00
DIETHYL ETHER	60-29-7	10.00	70.00	130.00		30.00
DI-ISOPROPYL ETHER	108-20-3	10.00	70.00	130.00		30.00
ETHYLBENZENE	100-41-4	10.00	75.00	125.00		30.00
HEXACHLOROBUTADIENE	87-68-3	10.00	50.00	140.00		30.00
ISOPROPYLBENZENE	98-82-8	10.00	75.00	125.00		30.00
M,P-XYLENES	1330-20-7	10.00	70.00	130.00		30.00
METHYL TERT-BUTYL ETHER	1634-04-4	10.00	65.00	125.00		30.00
METHYLENE CHLORIDE	75-09-2	10.00	55.00	140.00		30.00
NAPHTHALENE	91-20-3	10.00	55.00	140.00		30.00
N-BUTYLBENZENE	104-51-8	10.00	70.00	135.00		30.00
N-PROPYLBENZENE	103-65-1	10.00	70.00	130.00		30.00
O-XYLENE	95-47-6	10.00	80.00	120.00		30.00
SEC-BUTYLBENZENE	135-98-8	10.00	70.00	125.00		30.00
STYRENE	100-42-5	10.00	65.00	135.00		30.00
TERT-BUTYLBENZENE	98-06-6	10.00	70.00	130.00		30.00
TETRACHLOROETHENE	127-18-4	10.00	45.00	150.00		30.00
TOLUENE	108-88-3	10.00	75.00	120.00		30.00
TRANS-1,2-DICHLOROETHENE	156-60-5	10.00	60.00	140.00		30.00
TRANS-1,3-DICHLOROPROPENE	10061-02-6	10.00	55.00	140.00		30.00
TRICHLOROETHENE	79-01-6	10.00	70.00	125.00		30.00
TRICHLOROFLUOROMETHANE	75-69-4	10.00	60.00	145.00		30.00
VINYL ACETATE	108-05-4	10.00	70.00	130.00		30.00
VINYL CHLORIDE	75-01-4	10.00	50.00	145.00		30.00

Analyte Name	Analyte Label (CAS)	Lower Rejection (%)	Lower Recovery (%)	Upper Recovery (%)	Upper Rejection (%)	RPD (%)
Method: 8260B	**Matrix: Water**					
QC Type: Surrogate						
1,2-DICHLOROETHANE-D4	17060-07-0	30.00	70	120		
4-Bromofluorobenzene	460-00-4	30.00	75	120		
DIBROMOFLUOROMETHANE	1868-53-7	30.00	85	115		
TOLUENE-D8	2037-26-5	30.00	85	120		
Method: 8260B High	**Matrix: Soil**					
QC Type: Laboratory_Control_Sample						
1,1,1,2-TETRACHLOROETHANE	630-20-6	10.00	75.00	125.00		30.00
1,1,1-TRICHLOROETHANE	71-55-6	10.00	70.00	135.00		30.00
1,1,2,2-TETRACHLOROETHANE	79-34-5	10.00	55.00	130.00		30.00
1,1,2-TRICHLOROETHANE	79-00-5	10.00	60.00	125.00		30.00
1,1-DICHLOROETHANE	75-34-3	10.00	75.00	125.00		30.00
1,1-DICHLOROETHENE	75-35-4	10.00	65.00	135.00		30.00
1,1-DICHLOROPROPENE	563-58-6	10.00	70.00	135.00		30.00
1,2,3-TRICHLOROBENZENE	87-61-6	10.00	60.00	135.00		30.00
1,2,3-TRICHLOROPROPANE	96-18-4	10.00	65.00	130.00		30.00
1,2,4-TRICHLOROBENZENE	120-82-1	10.00	65.00	130.00		30.00
1,2,4-TRIMETHYLBENZENE	95-63-6	10.00	65.00	135.00		30.00
1,2-DIBROMO-3-CHLOROPROPANE	96-12-8	10.00	40.00	135.00		30.00
1,2-DIBROMOETHANE	106-93-4	10.00	70.00	125.00		30.00
1,2-DICHLOROBENZENE	95-50-1	10.00	75.00	120.00		30.00
1,2-DICHLOROETHANE	107-06-2	10.00	70.00	135.00		30.00
1,2-DICHLOROPROPANE	78-87-5	10.00	70.00	120.00		30.00
1,3,5-TRIMETHYLBENZENE	108-67-8	10.00	65.00	135.00		30.00
1,3-DICHLOROBENZENE	541-73-1	10.00	70.00	125.00		30.00
1,3-DICHLOROPROPANE	142-28-9	10.00	75.00	125.00		30.00
1,4-DICHLOROBENZENE	106-46-7	10.00	75.00	125.00		30.00
1,4-DIOXANE	123-91-1	10.00	70.00	130.00		20.00
2,2-DICHLOROPROPANE	594-20-7	10.00	65.00	135.00		30.00
2-BUTANONE	78-93-3	10.00	30.00	160.00		30.00
2-CHLOROTOLUENE	95-49-8	10.00	70.00	130.00		30.00
2-HEXANONE	591-78-6	10.00	45.00	145.00		30.00

Analyte Name	Analyte Label (CAS)	Lower Rejection (%)	Lower Recovery (%)	Upper Recovery (%)	Upper Rejection (%)	RPD (%)
Method: 8260B High	**Matrix: Soil**					
QC Type: Laboratory_Control_Sample						
4-CHLOROTOLUENE	106-43-4	10.00	75.00	125.00		30.00
4-ISOPROPYLTOLUENE	99-87-6	10.00	75.00	135.00		30.00
4-METHYL-2-PENTANONE	108-10-1	10.00	45.00	145.00		30.00
ACETONE	67-64-1	10.00	20.00	160.00		30.00
BENZENE	71-43-2	10.00	75.00	125.00		30.00
BROMOBENZENE	108-86-1	10.00	65.00	120.00		30.00
BROMOCHLOROMETHANE	74-97-5	10.00	70.00	125.00		30.00
BROMODICHLOROMETHANE	75-27-4	10.00	70.00	130.00		30.00
BROMOFORM	75-25-2	10.00	55.00	135.00		30.00
BROMOMETHANE	74-83-9	10.00	30.00	160.00		30.00
CARBON DISULFIDE	75-15-0	10.00	45.00	160.00		30.00
CARBON TETRACHLORIDE	56-23-5	10.00	65.00	135.00		30.00
CHLOROBENZENE	108-90-7	10.00	75.00	125.00		30.00
CHLOROETHANE	75-00-3	10.00	40.00	155.00		30.00
CHLOROFORM	67-66-3	10.00	70.00	125.00		30.00
CHLOROMETHANE	74-87-3	10.00	50.00	130.00		30.00
CIS-1,2-DICHLOROETHENE	156-59-2	10.00	65.00	125.00		30.00
CIS-1,3-DICHLOROPROPENE	10061-01-5	10.00	70.00	125.00		30.00
DIBROMOCHLOROMETHANE	124-48-1	10.00	65.00	130.00		30.00
DIBROMOMETHANE	74-95-3	10.00	75.00	130.00		30.00
DICHLORODIFLUOROMETHANE	75-71-8	10.00	35.00	135.00		30.00
DIETHYL ETHER	60-29-7	10.00	70.00	130.00		30.00
DI-ISOPROPYL ETHER	108-20-3	10.00	70.00	130.00		30.00
ETHYLBENZENE	100-41-4	10.00	75.00	125.00		30.00
HEXACHLOROBUTADIENE	87-68-3	10.00	55.00	140.00		30.00
ISOPROPYLBENZENE	98-82-8	10.00	75.00	130.00		30.00
M,P-XYLENES	1330-20-7	10.00	80.00	125.00		30.00
METHYL TERT-BUTYL ETHER	1634-04-4	10.00	70.00	130.00		30.00
METHYLENE CHLORIDE	75-09-2	10.00	55.00	140.00		30.00
NAPHTHALENE	91-20-3	10.00	40.00	125.00		30.00
N-BUTYLBENZENE	104-51-8	10.00	65.00	140.00		30.00
N-PROPYLBENZENE	103-65-1	10.00	65.00	135.00		30.00

Analyte Name	Analyte Label (CAS)	Lower Rejection (%)	Lower Recovery (%)	Upper Recovery (%)	Upper Rejection (%)	RPD (%)
Method: 8260B High	**Matrix:** Soil					
QC Type: Laboratory_Control_Sample						
O-XYLENE	95-47-6	10.00	75.00	125.00		30.00
SEC-BUTYLBENZENE	135-98-8	10.00	65.00	130.00		30.00
STYRENE	100-42-5	10.00	75.00	125.00		30.00
TERT-BUTYLBENZENE	98-06-6	10.00	65.00	130.00		30.00
TETRACHLOROETHENE	127-18-4	10.00	65.00	140.00		30.00
TOLUENE	108-88-3	10.00	70.00	125.00		30.00
TRANS-1,2-DICHLOROETHENE	156-60-5	10.00	65.00	135.00		30.00
TRANS-1,3-DICHLOROPROPENE	10061-02-6	10.00	65.00	125.00		30.00
TRICHLOROETHENE	79-01-6	10.00	75.00	125.00		30.00
TRICHLOROFLUOROMETHANE	75-69-4	10.00	25.00	185.00		30.00
VINYL ACETATE	108-05-4	10.00	70.00	130.00		30.00
VINYL CHLORIDE	75-01-4	10.00	60.00	125.00		30.00
QC Type: Matrix_Spike						
1,1,1,2-TETRACHLOROETHANE	630-20-6	10.00	75.00	125.00		30.00
1,1,1-TRICHLOROETHANE	71-55-6	10.00	70.00	135.00		30.00
1,1,2,2-TETRACHLOROETHANE	79-34-5	10.00	55.00	130.00		30.00
1,1,2-TRICHLOROETHANE	79-00-5	10.00	60.00	125.00		30.00
1,1-DICHLOROETHANE	75-34-3	10.00	75.00	125.00		30.00
1,1-DICHLOROETHENE	75-35-4	10.00	65.00	135.00		30.00
1,1-DICHLOROPROPENE	563-58-6	10.00	70.00	135.00		30.00
1,2,3-TRICHLOROBENZENE	87-61-6	10.00	60.00	135.00		30.00
1,2,3-TRICHLOROPROPANE	96-18-4	10.00	65.00	130.00		30.00
1,2,4-TRICHLOROBENZENE	120-82-1	10.00	65.00	130.00		30.00
1,2,4-TRIMETHYLBENZENE	95-63-6	10.00	65.00	135.00		30.00
1,2-DIBROMO-3-CHLOROPROPANE	96-12-8	10.00	40.00	135.00		30.00
1,2-DIBROMOETHANE	106-93-4	10.00	70.00	125.00		30.00
1,2-DICHLOROBENZENE	95-50-1	10.00	75.00	120.00		30.00
1,2-DICHLOROETHANE	107-06-2	10.00	70.00	135.00		30.00
1,2-DICHLOROPROPANE	78-87-5	10.00	70.00	120.00		30.00
1,3,5-TRIMETHYLBENZENE	108-67-8	10.00	65.00	135.00		30.00
1,3-DICHLOROBENZENE	541-73-1	10.00	70.00	125.00		30.00

Analyte Name	Analyte Label (CAS)	Lower Rejection (%)	Lower Recovery (%)	Upper Recovery (%)	Upper Rejection (%)	RPD (%)
Method: 8260B High	**Matrix:** Soil					
QC Type: Matrix_Spike						
1,3-DICHLOROPROPANE	142-28-9	10.00	75.00	125.00		30.00
1,4-DICHLOROBENZENE	106-46-7	10.00	70.00	125.00		30.00
1,4-DIOXANE	123-91-1	10.00	70.00	130.00		20.00
2,2-DICHLOROPROPANE	594-20-7	10.00	65.00	135.00		30.00
2-BUTANONE	78-93-3	10.00	30.00	160.00		30.00
2-CHLOROTOLUENE	95-49-8	10.00	70.00	130.00		30.00
2-HEXANONE	591-78-6	10.00	45.00	145.00		30.00
4-CHLOROTOLUENE	106-43-4	10.00	75.00	125.00		30.00
4-ISOPROPYLTOLUENE	99-87-6	10.00	75.00	135.00		30.00
4-METHYL-2-PENTANONE	108-10-1	10.00	45.00	145.00		30.00
ACETONE	67-64-1	10.00	20.00	160.00		30.00
BENZENE	71-43-2	10.00	75.00	125.00		30.00
BROMOBENZENE	108-86-1	10.00	65.00	120.00		30.00
BROMOCHLOROMETHANE	74-97-5	10.00	70.00	125.00		30.00
BROMODICHLOROMETHANE	75-27-4	10.00	70.00	130.00		30.00
BROMOFORM	75-25-2	10.00	55.00	135.00		30.00
BROMOMETHANE	74-83-9	10.00	30.00	160.00		30.00
CARBON DISULFIDE	75-15-0	10.00	45.00	160.00		30.00
CARBON TETRACHLORIDE	56-23-5	10.00	65.00	135.00		30.00
CHLOROBENZENE	108-90-7	10.00	75.00	125.00		30.00
CHLOROETHANE	75-00-3	10.00	40.00	155.00		30.00
CHLOROFORM	67-66-3	10.00	70.00	125.00		30.00
CHLOROMETHANE	74-87-3	10.00	50.00	130.00		30.00
CIS-1,2-DICHLOROETHENE	156-59-2	10.00	65.00	125.00		30.00
CIS-1,3-DICHLOROPROPENE	10061-01-5	10.00	70.00	125.00		30.00
DIBROMOCHLOROMETHANE	124-48-1	10.00	65.00	130.00		30.00
DIBROMOMETHANE	74-95-3	10.00	75.00	130.00		30.00
DICHLORODIFLUOROMETHANE	75-71-8	10.00	35.00	135.00		30.00
DIETHYL ETHER	60-29-7	10.00	70.00	130.00		30.00
DI-ISOPROPYL ETHER	108-20-3	10.00	70.00	130.00		30.00
ETHYLBENZENE	100-41-4	10.00	75.00	125.00		30.00
HEXACHLOROBUTADIENE	87-68-3	10.00	55.00	140.00		30.00

Analyte Name	Analyte Label (CAS)	Lower Rejection (%)	Lower Recovery (%)	Upper Recovery (%)	Upper Rejection (%)	RPD (%)
Method: 8260B High	**Matrix: Soil**					
QC Type: Matrix_Spike						
ISOPROPYLBENZENE	98-82-8	10.00	75.00	130.00		30.00
M,P-XYLENES	1330-20-7	10.00	80.00	125.00		30.00
METHYL TERT-BUTYL ETHER	1634-04-4	10.00	70.00	130.00		30.00
METHYLENE CHLORIDE	75-09-2	10.00	55.00	140.00		30.00
NAPHTHALENE	91-20-3	10.00	40.00	125.00		30.00
N-BUTYLBENZENE	104-51-8	10.00	65.00	140.00		30.00
N-PROPYLBENZENE	103-65-1	10.00	65.00	135.00		30.00
O-XYLENE	95-47-6	10.00	75.00	125.00		30.00
SEC-BUTYLBENZENE	135-98-8	10.00	65.00	130.00		30.00
STYRENE	100-42-5	10.00	75.00	125.00		30.00
TERT-BUTYLBENZENE	98-06-6	10.00	65.00	130.00		30.00
TETRACHLOROETHENE	127-18-4	10.00	65.00	140.00		30.00
TOLUENE	108-88-3	10.00	70.00	125.00		30.00
TRANS-1,2-DICHLOROETHENE	156-60-5	10.00	65.00	135.00		30.00
TRANS-1,3-DICHLOROPROPENE	10061-02-6	10.00	65.00	125.00		30.00
TRICHLOROETHENE	79-01-6	10.00	75.00	125.00		30.00
TRICHLOROFLUOROMETHANE	75-69-4	10.00	25.00	185.00		30.00
VINYL ACETATE	108-05-4	10.00	70.00	130.00		30.00
VINYL CHLORIDE	75-01-4	10.00	60.00	125.00		30.00
QC Type: Surrogate						
1,2-DICHLOROETHANE-D4	17060-07-0	10.00	70	120		
4-BROMOFLUOROBENZENE (SURR)	460-00-4	10.00	85	120		
DIBROMOFLUOROMETHANE	1868-53-7	10.00	85	115		
TOLUENE-D8	2037-26-5	10.00	85	115		
Method: 8260C	**Matrix: Soil**					
QC Type: Laboratory_Control_Sample						
1,1,2,2-TETRACHLOROETHANE	79-34-5	10.00	55.00	130.00		30.00
1,1,2-TRICHLOROETHANE	79-00-5	10.00	60.00	125.00		30.00
1,1-DICHLOROETHANE	75-34-3	10.00	75.00	125.00		30.00
1,1-DICHLOROETHENE	75-35-4	10.00	65.00	135.00		30.00

Analyte Name	Analyte Label (CAS)	Lower Rejection (%)	Lower Recovery (%)	Upper Recovery (%)	Upper Rejection (%)	RPD (%)
Method: 8260C	**Matrix:** Soil					
QC Type: Laboratory_Control_Sample						
1,2,4-TRIMETHYLBENZENE	95-63-6	10.00	65.00	135.00		30.00
1,2-DICHLOROETHANE	107-06-2	10.00	70.00	135.00		30.00
1,3,5-TRIMETHYLBENZENE	108-67-8	10.00	65.00	135.00		30.00
BENZENE	71-43-2	10.00	75.00	125.00		30.00
CHLOROFORM	67-66-3	10.00	70.0	135.0		30.0
CIS-1,2-DICHLOROETHENE	156-59-2	10.00	65.00	125.00		30.00
ETHYLBENZENE	100-41-4	10.00	75.00	125.00		30.00
m,p-Xylene	179601-23-1	10.00	80.00	125.00		30.00
NAPHTHALENE	91-20-3	10.00	55.0	140.0		30.0
O-XYLENE	95-47-6	10.00	75.00	125.00		30.00
TETRACHLOROETHENE	127-18-4	10.00	65.00	140.00		30.00
TOLUENE	108-88-3	10.00	70.00	125.00		30.00
TRANS-1,2-DICHLOROETHENE	156-60-5	10.00	65.00	135.00		30.00
TRICHLOROETHENE	79-01-6	10.00	75.00	125.00		30.00
VINYL CHLORIDE	75-01-4	10.00	60.00	125.00		30.00
QC Type: Matrix_Spike						
1,1,2,2-TETRACHLOROETHANE	79-34-5	0.00	55.00	130.00		30.00
1,1,2-TRICHLOROETHANE	79-00-5	0.00	60.00	125.00		30.00
1,1-DICHLOROETHANE	75-34-3	0.00	75.00	125.00		30.00
1,1-DICHLOROETHENE	75-35-4	0.00	65.00	135.00		30.00
1,2,4-TRIMETHYLBENZENE	95-63-6	0.00	65.00	135.00		30.00
1,2-DICHLOROETHANE	107-06-2	0.00	70.00	135.00		30.00
1,3,5-TRIMETHYLBENZENE	108-67-8	0.00	65.00	135.00		30.00
BENZENE	71-43-2	0.00	75.00	125.00		30.00
CHLOROFORM	67-66-3	0.00	70.0	135.0		30.0
CIS-1,2-DICHLOROETHENE	156-59-2	0.00	65.00	125.00		30.00
ETHYLBENZENE	100-41-4	0.00	75.00	125.00		30.00
m,p-Xylene	179601-23-1	0.00	80.00	125.00		30.00
NAPHTHALENE	91-20-3	0.00	55.0	140.0		30.0
O-XYLENE	95-47-6	0.00	75.00	125.00		30.00
TETRACHLOROETHENE	127-18-4	0.00	65.00	140.00		30.00

Analyte Name	Analyte Label (CAS)	Lower Rejection (%)	Lower Recovery (%)	Upper Recovery (%)	Upper Rejection (%)	RPD (%)
Method: 8260C **Matrix: Soil**						
QC Type: Matrix_Spike						
TOLUENE	108-88-3	0.00	70.00	125.00		30.00
TRANS-1,2-DICHLOROETHENE	156-60-5	0.00	65.00	135.00		30.00
TRICHLOROETHENE	79-01-6	0.00	75.00	125.00		30.00
VINYL CHLORIDE	75-01-4	0.00	60.00	125.00		30.00
QC Type: Surrogate						
4-Bromofluorobenzene	460-00-4	10.00	85	120		
Method: 9060 **Matrix: Water**						
QC Type: Laboratory_Control_Sample						
TOTAL ORGANIC CARBON (TOC)	TOC	50.00	85.00	115.00		
QC Type: Matrix_Spike						
TOTAL ORGANIC CARBON (TOC)	TOC	50.00	85.00	115.00		
Method: RSK175 **Matrix: Water**						
QC Type: Laboratory_Control_Sample						
ETHANE	74-84-0	10.00	60.00	140.00		30.00
ETHENE	74-85-1	10.00	60.00	140.00		30.00
METHANE	74-82-8	10.00	60.00	140.00		30.00
QC Type: Matrix_Spike						
ETHANE	74-84-0	10.00	60.00	140.00		30.00
ETHENE	74-85-1	10.00	60.00	140.00		30.00
METHANE	74-82-8	10.00	60.00	140.00		30.00
Method: TOC-LK **Matrix: Soil**						
QC Type: Laboratory_Control_Sample						
TOTAL ORGANIC CARBON (TOC)	TOC	10.00	80.00	120.00		

Analyte Name	Analyte Label (CAS)	Lower Rejection (%)	Lower Recovery (%)	Upper Recovery (%)	Upper Rejection (%)	RPD (%)
Method: TOC-LK **Matrix:** Soil						
QC Type: Matrix_Spike						
TOTAL ORGANIC CARBON (TOC)	TOC	10.00	75.00	125.00		

Project Target Analyte Reporting Limit, Blank Contamination, and Lab & Field Duplicate RPD Criteria

eQapp Name: Stone PR-58 02222013
Description:

Target Analyte Name	Analyte Label (CAS)	Quantitation Limit	Detection Limit	Units	Blank Contamination Rule	LabDup RPD	FieldDup RPD
Method: 5210B Matrix: Water							
BIOLOGIC OXYGEN DEMAND, FIVE-DAY	BOD5	3	3	mg/L	5.00	20.00	50.00
Method: 5220D Matrix: Water							
CHEMICAL OXYGEN DEMAND	COD	10	4	mg/L	5.00	20.00	50.00
Method: 524.2 Matrix: Water							
1,1,1,2-TETRACHLOROETHANE	630-20-6	0.5	0.07	ug/L	5.00	20.00	30.00
1,1,1-TRICHLOROETHANE	71-55-6	0.5	0.09	ug/L	5.00	20.00	30.00
1,1,2,2-TETRACHLOROETHANE	79-34-5	0.5	0.11	ug/L	5.00	20.00	30.00
1,1,2-TRICHLOROETHANE	79-00-5	0.5	0.14	ug/L	5.00	20.00	30.00
1,1-DICHLOROETHANE	75-34-3	0.5	0.09	ug/L	5.00	20.00	30.00
1,1-DICHLOROETHENE	75-35-4	0.5	0.15	ug/L	5.00	20.00	30.00
1,1-DICHLOROPROPENE	563-58-6	0.5	0.08	ug/L	5.00	20.00	30.00
1,2,3-TRICHLOROBENZENE	87-61-6	0.5	0.08	ug/L	5.00	20.00	30.00
1,2,3-TRICHLOROPROPANE	96-18-4	0.5	0.16	ug/L	5.00	20.00	30.00
1,2,4-TRICHLOROBENZENE	120-82-1	0.5	0.07	ug/L	5.00	20.00	30.00
1,2,4-TRIMETHYLBENZENE	95-63-6	0.5	0.07	ug/L	5.00	20.00	30.00
1,2-DIBROMO-3-CHLOROPROPANE	96-12-8	2.0	0.66	ug/L	5.00	20.00	30.00
1,2-DIBROMOETHANE	106-93-4	0.5	0.08	ug/L	5.00	20.00	30.00
1,2-DICHLOROBENZENE	95-50-1	0.5	0.07	ug/L	5.00	20.00	30.00
1,2-DICHLOROETHANE	107-06-2	0.5	0.13	ug/L	5.00	20.00	30.00
1,2-DICHLOROPROPANE	78-87-5	0.5	0.11	ug/L	5.00	20.00	30.00
1,3,5-TRIMETHYLBENZENE	108-67-8	0.5	0.07	ug/L	5.00	20.00	30.00
1,3-DICHLOROBENZENE	541-73-1	0.5	0.06	ug/L	5.00	20.00	30.00
1,3-DICHLOROPROPANE	142-28-9	0.5	0.10	ug/L	5.00	20.00	30.00
1,4-DICHLOROBENZENE	106-46-7	0.5	0.08	ug/L	5.00	20.00	30.00
1,4-DIOXANE	123-91-1	500	250	ug/L	5.00	20.00	30.00
2,2-DICHLOROPROPANE	594-20-7	0.5	0.12	ug/L	5.00	20.00	30.00
2-BUTANONE	78-93-3	5.0	1.60	ug/L	5.00	20.00	30.00

Target Analyte Name	Analyte Label (CAS)	Quantitation Limit	Detection Limit	Units	Blank Contamination Rule	LabDup RPD	FieldDup RPD
Method: 524.2 **Matrix: Water**							
2-CHLOROTOLUENE	95-49-8	0.5	0.10	ug/L	5.00	20.00	30.00
2-HEXANONE	591-78-6	5.0	1.03	ug/L	5.00	20.00	30.00
4-CHLOROTOLUENE	106-43-4	0.5	0.12	ug/L	5.00	20.00	30.00
4-METHYL-2-PENTANONE	108-10-1	5.0	0.98	ug/L	5.00	20.00	30.00
ACETONE	67-64-1	5.0	1.66	ug/L	5.00	20.00	30.00
BENZENE	71-43-2	0.5	0.06	ug/L	5.00	20.00	30.00
BROMOBENZENE	108-86-1	0.5	0.06	ug/L	5.00	20.00	30.00
BROMOCHLOROMETHANE	74-97-5	0.5	0.11	ug/L	5.00	20.00	30.00
BROMODICHLOROMETHANE	75-27-4	0.5	0.08	ug/L	5.00	20.00	30.00
BROMOFORM	75-25-2	0.5	0.16	ug/L	5.00	20.00	30.00
BROMOMETHANE	74-83-9	0.5	0.11	ug/L	5.00	20.00	30.00
CARBON DISULFIDE	75-15-0	0.5	0.09	ug/L	5.00	20.00	30.00
CARBON TETRACHLORIDE	56-23-5	0.5	0.09	ug/L	5.00	20.00	30.00
CHLOROBENZENE	108-90-7	0.5	0.06	ug/L	5.00	20.00	30.00
CHLOROETHANE	75-00-3	0.5	0.12	ug/L	5.00	20.00	30.00
CHLOROFORM	67-66-3	0.5	0.08	ug/L	5.00	20.00	30.00
CHLOROMETHANE	74-87-3	0.5	0.10	ug/L	5.00	20.00	30.00
CIS-1,2-DICHLOROETHENE	156-59-2	0.5	0.09	ug/L	5.00	20.00	30.00
CIS-1,3-DICHLOROPROPENE	10061-01-5	0.5	0.10	ug/L	5.00	20.00	30.00
DIBROMOCHLOROMETHANE	124-48-1	0.5	0.10	ug/L	5.00	20.00	30.00
DIBROMOMETHANE	74-95-3	0.5	0.10	ug/L	5.00	20.00	30.00
DICHLORODIFLUOROMETHANE	75-71-8	0.5	0.09	ug/L	5.00	20.00	30.00
DIETHYL ETHER	60-29-7	0.5	0.17	ug/L	5.00	20.00	30.00
DI-ISOPROPYL ETHER	108-20-3	0.5	0.14	ug/L	5.00	20.00	30.00
ETHYLBENZENE	100-41-4	0.5	0.07	ug/L	5.00	20.00	30.00
HEXACHLOROBUTADIENE	87-68-3	0.5	0.12	ug/L	5.00	20.00	30.00
ISOPROPYLBENZENE	98-82-8	0.5	0.06	ug/L	5.00	20.00	30.00
M,P-XYLENES	1330-20-7	1.0	0.14	ug/L	5.00	20.00	30.00
METHYL TERT-BUTYL ETHER	1634-04-4	0.5	0.10	ug/L	5.00	20.00	30.00
METHYLENE CHLORIDE	75-09-2	0.5	0.10	ug/L	10.00	20.00	30.00
NAPHTHALENE	91-20-3	0.5	0.16	ug/L	5.00	20.00	30.00
N-BUTYLBENZENE	104-51-8	0.5	0.06	ug/L	5.00	20.00	30.00
N-PROPYLBENZENE	103-65-1	0.5	0.10	ug/L	5.00	20.00	30.00

Target Analyte Name	Analyte Label (CAS)	Quantitation Limit	Detection Limit	Units	Blank Contamination Rule	LabDup RPD	FieldDup RPD
Method: 524.2 **Matrix: Water**							
O-XYLENE	95-47-6	0.5	0.07	ug/L	5.00	20.00	30.00
P-ISOPROPYLTOLUENE	99-87-6	0.5	0.07	ug/L	5.00	20.00	30.00
SEC-BUTYLBENZENE	135-98-8	0.5	0.08	ug/L	5.00	20.00	30.00
STYRENE	100-42-5	0.5	0.06	ug/L	5.00	20.00	30.00
TERT-BUTYLBENZENE	98-06-6	0.5	0.09	ug/L	5.00	20.00	30.00
TETRACHLOROETHENE	127-18-4	0.5	0.18	ug/L	5.00	20.00	30.00
TOLUENE	108-88-3	0.5	0.06	ug/L	5.00	20.00	30.00
TRANS-1,2-DICHLOROETHENE	156-60-5	0.5	0.07	ug/L	5.00	20.00	30.00
TRANS-1,3-DICHLOROPROPENE	10061-02-6	0.5	0.10	ug/L	5.00	20.00	30.00
TRICHLOROETHENE	79-01-6	0.5	0.10	ug/L	5.00	20.00	30.00
TRICHLOROFLUOROMETHANE	75-69-4	0.5	0.09	ug/L	5.00	20.00	30.00
VINYL ACETATE	108-05-4	5.0	0.31	ug/L	5.00	20.00	30.00
VINYL CHLORIDE	75-01-4	0.2	0.06	ug/L	5.00	20.00	30.00
Method: 6010B **Matrix: Water**							
ALUMINUM	7429-90-5	50.0	13.0	ug/L	5.00	20.00	50.00
BARIUM	7440-39-3	25.0	1.5	ug/L	5.00	20.00	50.00
BERYLLIUM	7440-41-7	0.5	0.1	ug/L	5.00	20.00	50.00
CADMIUM	7440-43-9	2.5	0.5	ug/L	5.00	20.00	50.00
CALCIUM	7440-70-2	100	32.5	ug/L	5.00	20.00	50.00
CHROMIUM	7440-47-3	10.0	1.5	ug/L	5.00	20.00	50.00
COBALT	7440-48-4	10.0	1.0	ug/L	5.00	20.00	50.00
COPPER	7440-50-8	10.0	2.0	ug/L	5.00	20.00	50.00
IRON	7439-89-6	50.0	11.5	ug/L	5.00	20.00	50.00
LEAD	7439-92-1	10.0	2.5	ug/L	5.00	20.00	50.00
MAGNESIUM	7439-95-4	100.0	21.0	ug/L	5.00	20.00	50.00
MANGANESE	7439-96-5	10.0	3.0	ug/L	5.00	20.00	50.00
NICKEL	7440-02-0	25.0	1.0	ug/L	5.00	20.00	50.00
POTASSIUM	7440-09-7	2500	55.0	ug/L	5.00	20.00	50.00
SELENIUM	7782-49-2	25.0	8.5	ug/L	5.00	20.00	50.00
SILVER	7440-22-4	5.0	1.0	ug/L	5.00	20.00	50.00
VANADIUM	7440-62-2	10.0	1.0	ug/L	5.00	20.00	50.00
ZINC	7440-66-6	25.0	4.5	ug/L	5.00	20.00	50.00

Target Analyte Name	Analyte Label (CAS)	Quantitation Limit	Detection Limit	Units	Blank Contamination Rule	LabDup RPD	FieldDup RPD
Method: 6010B-Ca **Matrix: Water**							
HARDNESS (AS CACO3)	HARD	1500	335	ug/L	5.00	20.00	50.00
Method: 7041 **Matrix: Water**							
ANTIMONY	7440-36-0	2.5	0.6	ug/L	5.00	20.00	50.00
Method: 7060A **Matrix: Water**							
ARSENIC	7440-38-2	2.5	0.4	ug/L	5.00	20.00	50.00
Method: 7470A **Matrix: Water**							
MERCURY	7439-97-6	0.5	0.12	ug/L	5.00	20.00	50.00
Method: 7841 **Matrix: Water**							
THALLIUM	7440-28-0	1.5	0.4	ug/L	5.00	20.00	50.00
Method: 8100M **Matrix: Soil**							
PETROLEUM HYDROCARBONS (TPH)	PHC	37.5	5.6	mg/kg	5.00	50.00	50.00
Method: 8260B **Matrix: Soil**							
1,1,1,2-TETRACHLOROETHANE	630-20-6	0.005	0.0015	mg/Kg	5.00	20.00	50.00
1,1,1-TRICHLOROETHANE	71-55-6	0.005	0.0016	mg/Kg	5.00	20.00	50.00
1,1,2,2-TETRACHLOROETHANE	79-34-5	0.005	0.0015	mg/Kg	5.00	20.00	50.00
1,1,2-TRICHLOROETHANE	79-00-5	0.005	0.0017	mg/Kg	5.00	20.00	50.00
1,1-DICHLOROETHANE	75-34-3	0.005	0.0016	mg/Kg	5.00	20.00	50.00
1,1-DICHLOROETHENE	75-35-4	0.005	0.0016	mg/Kg	5.00	20.00	50.00
1,1-DICHLOROPROPENE	563-58-6	0.005	0.0016	mg/Kg	5.00	20.00	50.00
1,2,3-TRICHLOROBENZENE	87-61-6	0.005	0.0014	mg/Kg	5.00	20.00	50.00
1,2,3-TRICHLOROPROPANE	96-18-4	0.005	0.0017	mg/Kg	5.00	20.00	50.00
1,2,4-TRICHLOROBENZENE	120-82-1	0.005	0.0013	mg/Kg	5.00	20.00	50.00
1,2,4-TRIMETHYLBENZENE	95-63-6	0.005	0.0016	mg/Kg	5.00	20.00	50.00
1,2-DIBROMO-3-CHLOROPROPANE	96-12-8	0.005	0.0016	mg/Kg	5.00	20.00	50.00
1,2-DIBROMOETHANE	106-93-4	0.005	0.0015	mg/Kg	5.00	20.00	50.00
1,2-DICHLOROBENZENE	95-50-1	0.005	0.0015	mg/Kg	5.00	20.00	50.00
1,2-DICHLOROETHANE	107-06-2	0.005	0.0016	mg/Kg	5.00	20.00	50.00
1,2-DICHLOROPROPANE	78-87-5	0.005	0.0017	mg/Kg	5.00	20.00	50.00
1,3,5-TRIMETHYLBENZENE	108-67-8	0.005	0.0016	mg/Kg	5.00	20.00	50.00
1,3-DICHLOROBENZENE	541-73-1	0.005	0.0016	mg/Kg	5.00	20.00	50.00

Target Analyte Name	Analyte Label (CAS)	Quantitation Limit	Detection Limit	Units	Blank Contamination Rule	LabDup RPD	FieldDup RPD
Method: 8260B **Matrix: Soil**							
1,3-DICHLOROPROPANE	142-28-9	0.005	0.0015	mg/Kg	5.00	20.00	50.00
1,4-DICHLOROBENZENE	106-46-7	0.005	0.0015	mg/Kg	5.00	20.00	50.00
1,4-DIOXANE	123-91-1	0.100	0.010	mg/Kg	5.00	20.00	50.00
2,2-DICHLOROPROPANE	594-20-7	0.005	0.0016	mg/Kg	5.00	20.00	50.00
2-BUTANONE	78-93-3	0.050	0.0024	mg/Kg	5.00	20.00	50.00
2-CHLOROTOLUENE	95-49-8	0.005	0.0010	mg/Kg	5.00	20.00	50.00
2-HEXANONE	591-78-6	0.050	0.0036	mg/Kg	5.00	20.00	50.00
4-CHLOROTOLUENE	106-43-4	0.005	0.0017	mg/Kg	5.00	20.00	50.00
4-ISOPROPYLTOLUENE	99-87-6	0.005	0.0016	mg/Kg	5.00	20.00	50.00
4-METHYL-2-PENTANONE	108-10-1	0.050	0.0013	mg/Kg	5.00	20.00	50.00
ACETONE	67-64-1	0.050	0.0031	mg/Kg	5.00	20.00	50.00
BENZENE	71-43-2	0.005	0.0017	mg/Kg	5.00	20.00	50.00
BROMOBENZENE	108-86-1	0.005	0.0016	mg/Kg	5.00	20.00	50.00
BROMOCHLOROMETHANE	74-97-5	0.005	0.0016	mg/Kg	5.00	20.00	50.00
BROMODICHLOROMETHANE	75-27-4	0.005	0.0016	mg/Kg	5.00	20.00	50.00
BROMOFORM	75-25-2	0.005	0.0014	mg/Kg	5.00	20.00	50.00
BROMOMETHANE	74-83-9	0.010	0.0017	mg/Kg	5.00	20.00	50.00
CARBON DISULFIDE	75-15-0	0.005	0.0016	mg/Kg	5.00	20.00	50.00
CARBON TETRACHLORIDE	56-23-5	0.005	0.0016	mg/Kg	5.00	20.00	50.00
CHLOROBENZENE	108-90-7	0.005	0.0016	mg/Kg	5.00	20.00	50.00
CHLOROETHANE	75-00-3	0.010	0.0018	mg/Kg	5.00	20.00	50.00
CHLOROFORM	67-66-3	0.005	0.0017	mg/Kg	5.00	20.00	50.00
CHLOROMETHANE	74-87-3	0.010	0.0017	mg/Kg	5.00	20.00	50.00
CIS-1,2-DICHLOROETHENE	156-59-2	0.005	0.0017	mg/Kg	5.00	20.00	50.00
CIS-1,3-DICHLOROPROPENE	10061-01-5	0.005	0.0017	mg/Kg	5.00	20.00	50.00
DIBROMOCHLOROMETHANE	124-48-1	0.005	0.0016	mg/Kg	5.00	20.00	50.00
DIBROMOMETHANE	74-95-3	0.005	0.0017	mg/Kg	5.00	20.00	50.00
DICHLORODIFLUOROMETHANE	75-71-8	0.010	0.0014	mg/Kg	5.00	20.00	50.00
DIETHYL ETHER	60-29-7	0.005	0.0017	mg/Kg	5.00	20.00	50.00
DI-ISOPROPYL ETHER	108-20-3	0.005	0.0016	mg/Kg	5.00	20.00	50.00
ETHYLBENZENE	100-41-4	0.005	0.0016	mg/Kg	5.00	20.00	50.00
HEXACHLOROBUTADIENE	87-68-3	0.005	0.0015	mg/Kg	5.00	20.00	50.00
ISOPROPYLBENZENE	98-82-8	0.005	0.0017	mg/Kg	5.00	20.00	50.00

Target Analyte Name	Analyte Label (CAS)	Quantitation Limit	Detection Limit	Units	Blank Contamination Rule	LabDup RPD	FieldDup RPD
Method: 8260B **Matrix: Soil**							
M,P-XYLENES	1330-20-7	0.010	0.0032	mg/Kg	5.00	20.00	50.00
METHYL TERT-BUTYL ETHER	1634-04-4	0.005	0.0017	mg/Kg	5.00	20.00	50.00
METHYLENE CHLORIDE	75-09-2	0.025	0.0019	mg/Kg	5.00	20.00	50.00
NAPHTHALENE	91-20-3	0.005	0.0013	mg/Kg	5.00	20.00	50.00
N-BUTYLBENZENE	104-51-8	0.005	0.0016	mg/Kg	5.00	20.00	50.00
N-PROPYLBENZENE	103-65-1	0.005	0.0012	mg/Kg	5.00	20.00	50.00
O-XYLENE	95-47-6	0.005	0.0016	mg/Kg	5.00	20.00	50.00
SEC-BUTYLBENZENE	135-98-8	0.005	0.0016	mg/Kg	5.00	20.00	50.00
STYRENE	100-42-5	0.005	0.0015	mg/Kg	5.00	20.00	50.00
TERT-BUTYLBENZENE	98-06-6	0.005	0.0016	mg/Kg	5.00	20.00	50.00
TETRACHLOROETHENE	127-18-4	0.005	0.0017	mg/Kg	5.00	20.00	50.00
TOLUENE	108-88-3	0.005	0.0016	mg/Kg	5.00	20.00	50.00
TRANS-1,2-DICHLOROETHENE	156-60-5	0.005	0.0017	mg/Kg	5.00	20.00	50.00
TRANS-1,3-DICHLOROPROPENE	10061-02-6	0.005	0.0016	mg/Kg	5.00	20.00	50.00
TRICHLOROETHENE	79-01-6	0.005	0.0017	mg/Kg	5.00	20.00	50.00
TRICHLOROFLUOROMETHANE	75-69-4	0.005	0.0017	mg/Kg	5.00	20.00	50.00
VINYL ACETATE	108-05-4	0.005		mg/Kg	5.00	20.00	50.00
VINYL CHLORIDE	75-01-4	0.010	0.0017	mg/Kg	5.00	20.00	50.00
Method: 8260B **Matrix: Water**							
1,1,1,2-TETRACHLOROETHANE	630-20-6	1.0	0.2	ug/L	5.00		50.00
1,1,1-TRICHLOROETHANE	71-55-6	1.0	0.2	ug/L	5.00		50.00
1,1,2,2-TETRACHLOROETHANE	79-34-5	0.5	0.1	ug/L	5.00		50.00
1,1,2-TRICHLOROETHANE	79-00-5	1.0	0.2	ug/L	5.00		50.00
1,1-DICHLOROETHANE	75-34-3	1.0	0.2	ug/L	5.00		50.00
1,1-DICHLOROETHENE	75-35-4	1.0	0.3	ug/L	5.00		50.00
1,1-DICHLOROPROPENE	563-58-6	2.0	0.2	ug/L	5.00		50.00
1,2,3-TRICHLOROBENZENE	87-61-6	1.0	0.2	ug/L	5.00		50.00
1,2,3-TRICHLOROPROPANE	96-18-4	1.0	0.3	ug/L	5.00		50.00
1,2,4-TRICHLOROBENZENE	120-82-1	1.0	0.2	ug/L	5.00		50.00
1,2,4-TRIMETHYLBENZENE	95-63-6	1.0	0.1	ug/L	5.00		50.00
1,2-DIBROMO-3-CHLOROPROPANE	96-12-8	5.0	1.0	ug/L	5.00		50.00
1,2-DIBROMOETHANE	106-93-4	1.0	0.2	ug/L	5.00		50.00

Target Analyte Name	Analyte Label (CAS)	Quantitation Limit	Detection Limit	Units	Blank Contamination Rule	LabDup RPD	FieldDup RPD
Method: 8260B **Matrix: Water**							
1,2-DICHLOROBENZENE	95-50-1	1.0	0.1	ug/L	5.00		50.00
1,2-DICHLOROETHANE	107-06-2	1.0	0.2	ug/L	5.00		50.00
1,2-DICHLOROPROPANE	78-87-5	1.0	0.2	ug/L	5.00		50.00
1,3,5-TRIMETHYLBENZENE	108-67-8	1.0	0.1	ug/L	5.00		50.00
1,3-DICHLOROBENZENE	541-73-1	1.0	0.2	ug/L	5.00		50.00
1,3-DICHLOROPROPANE	142-28-9	1.0	0.1	ug/L	5.00		50.00
1,4-DICHLOROBENZENE	106-46-7	1.0	0.1	ug/L	5.00		50.00
1,4-DIOXANE	123-91-1	500	190	ug/L	5.00		50.00
2,2-DICHLOROPROPANE	594-20-7	1.0	0.3	ug/L	5.00		50.00
2-BUTANONE	78-93-3	10.0	3.4	ug/L	10.00		50.00
2-CHLOROTOLUENE	95-49-8	1.0	0.1	ug/L	5.00		50.00
2-HEXANONE	591-78-6	10.0	1.5	ug/L	5.00		50.00
4-CHLOROTOLUENE	106-43-4	1.0	0.1	ug/L	5.00		50.00
4-ISOPROPYLTOLUENE	99-87-6	1.0	0.1	ug/L	5.00		50.00
4-METHYL-2-PENTANONE	108-10-1	10.0	1.6	ug/L	5.00		50.00
ACETONE	67-64-1	10.0	2.7	ug/L	10.00		50.00
BENZENE	71-43-2	1.0	0.1	ug/L	5.00		50.00
BROMOBENZENE	108-86-1	2.0	0.2	ug/L	5.00		50.00
BROMOCHLOROMETHANE	74-97-5	1.0	0.3	ug/L	5.00		50.00
BROMODICHLOROMETHANE	75-27-4	0.6	0.1	ug/L	5.00		50.00
BROMOFORM	75-25-2	1.0	0.2	ug/L	5.00		50.00
BROMOMETHANE	74-83-9	2.0	0.4	ug/L	5.00		50.00
CARBON DISULFIDE	75-15-0	1.0	0.1	ug/L	10.00		50.00
CARBON TETRACHLORIDE	56-23-5	1.0	0.1	ug/L	5.00		50.00
CHLOROBENZENE	108-90-7	1.0	0.1	ug/L	5.00		50.00
CHLOROETHANE	75-00-3	2.0	0.4	ug/L	5.00		50.00
CHLOROFORM	67-66-3	1.0	0.2	ug/L	5.00		50.00
CHLOROMETHANE	74-87-3	2.0	0.2	ug/L	5.00		50.00
CIS-1,2-DICHLOROETHENE	156-59-2	1.0	0.2	ug/L	5.00		50.00
CIS-1,3-DICHLOROPROPENE	10061-01-5	0.4	0.2	ug/L	5.00		50.00
DIBROMOCHLOROMETHANE	124-48-1	1.0	0.2	ug/L	5.00		50.00
DIBROMOMETHANE	74-95-3	1.0	0.3	ug/L	5.00		50.00
DICHLORODIFLUOROMETHANE	75-71-8	2.0	0.3	ug/L	5.00		50.00

Target Analyte Name	Analyte Label (CAS)	Quantitation Limit	Detection Limit	Units	Blank Contamination Rule	LabDup RPD	FieldDup RPD
Method: 8260B **Matrix: Water**							
DIETHYL ETHER	60-29-7	1.0	0.2	ug/L	5.00		50.00
DI-ISOPROPYL ETHER	108-20-3	1.0	0.3	ug/L	5.00		50.00
ETHYLBENZENE	100-41-4	1.0	0.1	ug/L	5.00		50.00
HEXACHLOROBUTADIENE	87-68-3	0.6	0.2	ug/L	5.00		50.00
ISOPROPYLBENZENE	98-82-8	1.0	0.1	ug/L	5.00		50.00
M,P-XYLENES	1330-20-7	2.0	0.2	ug/L	5.00		50.00
METHYL TERT-BUTYL ETHER	1634-04-4	1.0	0.3	ug/L	5.00		50.00
METHYLENE CHLORIDE	75-09-2	2.0	0.2	ug/L	10.00		50.00
NAPHTHALENE	91-20-3	1.0	0.2	ug/L	5.00		50.00
N-BUTYLBENZENE	104-51-8	1.0	0.1	ug/L	5.00		50.00
N-PROPYLBENZENE	103-65-1	1.0	0.2	ug/L	5.00		50.00
O-XYLENE	95-47-6	1.0	0.1	ug/L	5.00		50.00
SEC-BUTYLBENZENE	135-98-8	1.0	0.1	ug/L	5.00		50.00
STYRENE	100-42-5	1.0	0.1	ug/L	5.00		50.00
TERT-BUTYLBENZENE	98-06-6	1.0	0.1	ug/L	5.00		50.00
TETRACHLOROETHENE	127-18-4	1.0	0.2	ug/L	5.00		50.00
TOLUENE	108-88-3	1.0	0.1	ug/L	5.00		50.00
TRANS-1,2-DICHLOROETHENE	156-60-5	1.0	0.3	ug/L	5.00		50.00
TRANS-1,3-DICHLOROPROPENE	10061-02-6	0.4	0.2	ug/L	5.00		50.00
TRICHLOROETHENE	79-01-6	1.0	0.2	ug/L	5.00		50.00
TRICHLOROFLUOROMETHANE	75-69-4	1.0	0.4	ug/L	5.00		50.00
VINYL ACETATE	108-05-4	5.0	0.5	ug/L	5.00		50.00
VINYL CHLORIDE	75-01-4	1.0	0.2	ug/L	5.00		50.00
Method: 8260B High **Matrix: Soil**							
1,1,1,2-TETRACHLOROETHANE	630-20-6	0.100	0.0087	mg/Kg	5.00	20.00	50.00
1,1,1-TRICHLOROETHANE	71-55-6	0.050	0.0088	mg/Kg	5.00	20.00	50.00
1,1,2,2-TETRACHLOROETHANE	79-34-5	0.050	0.0136	mg/Kg	5.00	20.00	50.00
1,1,2-TRICHLOROETHANE	79-00-5	0.050	0.0125	mg/Kg	5.00	20.00	50.00
1,1-DICHLOROETHANE	75-34-3	0.050	0.0080	mg/Kg	5.00	20.00	50.00
1,1-DICHLOROETHENE	75-35-4	0.050	0.0123	mg/Kg	5.00	20.00	50.00
1,1-DICHLOROPROPENE	563-58-6	0.050	0.0077	mg/Kg	5.00	20.00	50.00
1,2,3-TRICHLOROBENZENE	87-61-6	0.050	0.0167	mg/Kg	5.00	20.00	50.00

Target Analyte Name	Analyte Label (CAS)	Quantitation Limit	Detection Limit	Units	Blank Contamination Rule	LabDup RPD	FieldDup RPD
Method: 8260B High **Matrix: Soil**							
1,2,3-TRICHLOROPROPANE	96-18-4	0.050	0.0124	mg/Kg	5.00	20.00	50.00
1,2,4-TRICHLOROBENZENE	120-82-1	0.050	0.0110	mg/Kg	5.00	20.00	50.00
1,2,4-TRIMETHYLBENZENE	95-63-6	0.050	0.0096	mg/Kg	5.00	20.00	50.00
1,2-DIBROMO-3-CHLOROPROPANE	96-12-8	0.300	0.1000	mg/Kg	5.00	20.00	50.00
1,2-DIBROMOETHANE	106-93-4	0.050	0.0127	mg/Kg	5.00	20.00	50.00
1,2-DICHLOROBENZENE	95-50-1	0.050	0.0071	mg/Kg	5.00	20.00	50.00
1,2-DICHLOROETHANE	107-06-2	0.050	0.0134	mg/Kg	5.00	20.00	50.00
1,2-DICHLOROPROPANE	78-87-5	0.050	0.0131	mg/Kg	5.00	20.00	50.00
1,3,5-TRIMETHYLBENZENE	108-67-8	0.050	0.0088	mg/Kg	5.00	20.00	50.00
1,3-DICHLOROBENZENE	541-73-1	0.050	0.0063	mg/Kg	5.00	20.00	50.00
1,3-DICHLOROPROPANE	142-28-9	0.050	0.0112	mg/Kg	5.00	20.00	50.00
1,4-DICHLOROBENZENE	106-46-7	0.050	0.0133	mg/Kg	5.00	20.00	50.00
1,4-DIOXANE	123-91-1	5.00	1.67	mg/Kg	5.00	20.00	50.00
2,2-DICHLOROPROPANE	594-20-7	0.100	0.0171	mg/Kg	5.00	20.00	50.00
2-BUTANONE	78-93-3	1.25	0.289	mg/Kg	5.00	20.00	50.00
2-CHLOROTOLUENE	95-49-8	0.050	0.0141	mg/Kg	5.00	20.00	50.00
2-HEXANONE	591-78-6	0.500	0.0861	mg/Kg	5.00	20.00	50.00
4-CHLOROTOLUENE	106-43-4	0.050	0.0065	mg/Kg	5.00	20.00	50.00
4-ISOPROPYLTOLUENE	99-87-6	0.050	0.0089	mg/Kg	5.00	20.00	50.00
4-METHYL-2-PENTANONE	108-10-1	0.500	0.0602	mg/Kg	5.00	20.00	50.00
ACETONE	67-64-1	1.25	0.37	mg/Kg	5.00	20.00	50.00
BENZENE	71-43-2	0.050	0.008	mg/Kg	5.00	20.00	50.00
BROMOBENZENE	108-86-1	0.050	0.0137	mg/Kg	5.00	20.00	50.00
BROMOCHLOROMETHANE	74-97-5	0.050	0.0162	mg/Kg	5.00	20.00	50.00
BROMODICHLOROMETHANE	75-27-4	0.050	0.0069	mg/Kg	5.00	20.00	50.00
BROMOFORM	75-25-2	0.050	0.0144	mg/Kg	5.00	20.00	50.00
BROMOMETHANE	74-83-9	0.100	0.0334	mg/Kg	5.00	20.00	50.00
CARBON DISULFIDE	75-15-0	0.050	0.0074	mg/Kg	5.00	20.00	50.00
CARBON TETRACHLORIDE	56-23-5	0.050	0.0087	mg/Kg	5.00	20.00	50.00
CHLOROBENZENE	108-90-7	0.050	0.0079	mg/Kg	5.00	20.00	50.00
CHLOROETHANE	75-00-3	0.100	0.0333	mg/Kg	5.00	20.00	50.00
CHLOROFORM	67-66-3	0.050	0.0103	mg/Kg	5.00	20.00	50.00
CHLOROMETHANE	74-87-3	0.100	0.0127	mg/Kg	5.00	20.00	50.00

Target Analyte Name	Analyte Label (CAS)	Quantitation Limit	Detection Limit	Units	Blank Contamination Rule	LabDup RPD	FieldDup RPD
Method: 8260B High **Matrix: Soil**							
CIS-1,2-DICHLOROETHENE	156-59-2	0.050	0.0124	mg/Kg	5.00	20.00	50.00
CIS-1,3-DICHLOROPROPENE	10061-01-5	0.050	0.0113	mg/Kg	5.00	20.00	50.00
DIBROMOCHLOROMETHANE	124-48-1	0.050	0.0126	mg/Kg	5.00	20.00	50.00
DIBROMOMETHANE	74-95-3	0.050	0.0158	mg/Kg	5.00	20.00	50.00
DICHLORODIFLUOROMETHANE	75-71-8	0.050	0.0087	mg/Kg	5.00	20.00	50.00
DIETHYL ETHER	60-29-7	0.050	0.0127	mg/Kg	5.00	20.00	50.00
DI-ISOPROPYL ETHER	108-20-3	0.050	0.0094	mg/Kg	5.00	20.00	50.00
ETHYLBENZENE	100-41-4	0.050	0.0065	mg/Kg	5.00	20.00	50.00
HEXACHLOROBUTADIENE	87-68-3	0.050	0.0167	mg/Kg	5.00	20.00	50.00
ISOPROPYLBENZENE	98-82-8	0.050	0.0088	mg/Kg	5.00	20.00	50.00
M,P-XYLENES	1330-20-7	0.100	0.0194	mg/Kg	5.00	20.00	50.00
METHYL TERT-BUTYL ETHER	1634-04-4	0.050	0.0080	mg/Kg	5.00	20.00	50.00
METHYLENE CHLORIDE	75-09-2	0.250	0.0131	mg/Kg	5.00	20.00	50.00
NAPHTHALENE	91-20-3	0.050	0.0131	mg/Kg	5.00	20.00	50.00
N-BUTYLBENZENE	104-51-8	0.050	0.0123	mg/Kg	5.00	20.00	50.00
N-PROPYLBENZENE	103-65-1	0.050	0.0122	mg/Kg	5.00	20.00	50.00
O-XYLENE	95-47-6	0.050	0.0096	mg/Kg	5.00	20.00	50.00
SEC-BUTYLBENZENE	135-98-8	0.050	0.0067	mg/Kg	5.00	20.00	50.00
STYRENE	100-42-5	0.050	0.0066	mg/Kg	5.00	20.00	50.00
TERT-BUTYLBENZENE	98-06-6	0.050	0.0117	mg/Kg	5.00	20.00	50.00
TETRACHLOROETHENE	127-18-4	0.050	0.0167	mg/Kg	5.00	20.00	50.00
TOLUENE	108-88-3	0.050	0.0127	mg/Kg	5.00	20.00	50.00
TRANS-1,2-DICHLOROETHENE	156-60-5	0.050	0.0164	mg/Kg	5.00	20.00	50.00
TRANS-1,3-DICHLOROPROPENE	10061-02-6	0.050	0.0154	mg/Kg	5.00	20.00	50.00
TRICHLOROETHENE	79-01-6	0.050	0.0103	mg/Kg	5.00	20.00	50.00
TRICHLOROFLUOROMETHANE	75-69-4	0.050	0.0132	mg/Kg	5.00	20.00	50.00
VINYL ACETATE	108-05-4	0.250	0.0103	mg/Kg	5.00	20.00	50.00
VINYL CHLORIDE	75-01-4	0.050	0.0165	mg/Kg	5.00	20.00	50.00
Method: 8260C **Matrix: Soil**							
1,1,2,2-TETRACHLOROETHANE	79-34-5	80	22	ug/Kg	5.00		50.00
1,1,2-TRICHLOROETHANE	79-00-5	80	20	ug/Kg	5.00		50.00
1,1-DICHLOROETHANE	75-34-3	80	15	ug/Kg	5.00		50.00

Target Analyte Name	Analyte Label (CAS)	Quantitation Limit	Detection Limit	Units	Blank Contamination Rule	LabDup RPD	FieldDup RPD
Method: 8260C **Matrix: Soil**							
1,1-DICHLOROETHENE	75-35-4	80	16	ug/Kg	5.00		50.00
1,2,4-TRIMETHYLBENZENE	95-63-6	80	23	ug/Kg	5.00		50.00
1,2-DICHLOROETHANE	107-06-2	80	17	ug/Kg	5.00		50.00
1,3,5-TRIMETHYLBENZENE	108-67-8	80	22	ug/Kg	5.00		50.00
BENZENE	71-43-2	80	5.2	ug/Kg	5.00		50.00
CHLOROFORM	67-66-3	80	9.1	ug/Kg	5.0		50.0
CIS-1,2-DICHLOROETHENE	156-59-2	80	8.4	ug/Kg	5.00		50.00
ETHYLBENZENE	100-41-4	80	24	ug/Kg	5.00		50.00
m,p-Xylene	179601-23-1	80	22	ug/Kg	5.00		50.00
NAPHTHALENE	91-20-3	80	25	ug/Kg	5.0		50.00
O-XYLENE	95-47-6	80	22	ug/Kg	5.00		50.00
TETRACHLOROETHENE	127-18-4	80	27	ug/Kg	5.00		50.00
TOLUENE	108-88-3	80	7.2	ug/Kg	5.00		50.00
TRANS-1,2-DICHLOROETHENE	156-60-5	80	7.7	ug/Kg	5.00		50.00
TRICHLOROETHENE	79-01-6	80	8.0	ug/Kg	5.00		50.00
VINYL CHLORIDE	75-01-4	80	13	ug/Kg	5.00		50.00
Method: 9060 **Matrix: Water**							
TOTAL ORGANIC CARBON (TOC)	TOC	1.0	1.0	mg/L	5.00	20.00	50.00
Method: ASTM D7262-10 **Matrix: Soil**							
PERCENT ASH	PERASH	0.1	0.1	%	5.00	30.00	50.00
PERCENT MOISTURE	PERMOIST	0.1	0.1	%	5.00	30.00	50.00
PERCENT ORGANIC MATTER	PERORG	0.1	0.1	%	5.00	30.00	50.00
PERCENT SOLIDS	PERSOLIDS	0.1	0.1	%	5.00	30.00	50.00
Method: RSK175 **Matrix: Water**							
ETHANE	74-84-0	4.8	0.9	ug/L	5.00	20.00	50.00
ETHENE	74-85-1	6.6	1.4	ug/L	5.00	20.00	50.00
METHANE	74-82-8	2.4	0.5	ug/L	5.00	20.00	50.00
Method: TOC-LK **Matrix: Soil**							
TOTAL ORGANIC CARBON (TOC)	TOC	200		mg/Kg	5.00	20.00	50.00

B: STANDARD OPERATING PROCEDURES

STANDARD OPERATING PROCEDURE

SEI-4.2.7

CHAIN OF CUSTODY PROCEDURES

SOP Number: SEI-4.2.7
Revision Number: 7

Date Issued: 04/09/93
Date of Revision: 5/10/12

1.0 OBJECTIVE

Chain of custody (COC) procedures documents the historical possession of sample containers and samples. Chain of custody records allow the possession and handling of samples to be tracked from the time of field collection until the samples are delivered to the laboratory. Every effort shall be made to keep as few people as possible in the chain of sample possession.

2.0 POLICIES

1. To maintain a record of sample collection, transfer between personnel, shipment, and receipt by the laboratory, a chain-of-custody record will be completed for each sample shipment by the field personnel or samplers.
2. Custody, as defined by this document, is referred as:
    - It is in someone's actual possession
    - It is in someone's view, after being in their physical possession, or
    - It was in someone's possession and then locked, sealed, or secured in a manner which prevents unsuspected tampering, or
    - It is placed in a designated and secured area
3. Upon receipt at the laboratory, it is the responsibility of the laboratory to note if tampering has occurred during sample shipment. If the coolers or containers are opened before receipt at the laboratory, the project manager will be contacted.

3.0 SAFETY ISSUES

1. The toxicity and carcinogenicity of each test substance used has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable.
2. All labels affixed to sampling containers must be carefully read and followed.

3. If necessary and appropriate, all chemicals are required to be received with a Material Safety Data Sheets (MSDS) or appropriate application label. These labels or MSDS shall be made available to all personnel involved in the sampling and testing.

4.0 PROCEDURES

4.1 Chain-of-Custody Record

The record of possession of the samples is kept on a COC form that accompanies each package of samples that are shipped to the lab. The exact format of the COC form may vary based on the sampling activities, but all forms must include the following information (example forms are included in Attachment 1):

1. Name, address, and if necessary, the telephone number of Stone Environmental, Inc.
2. Name (s) of the sampler (s).
3. Project or study designation.
4. Sample identification; if numerous samples are included in the package, a separate list of samples may be attached.
5. Date of sample collection (or dates, if samples in container were collected over more than one day).
6. Type of sample (water, soil, etc.).
7. Analyses to be performed.
8. The name and address of the analytical laboratory to which the samples are being shipped.
9. Signatures of all persons receiving/relinquishing the samples (does not include couriers).
10. Date and time of receipt and date and time of relinquishment for each person in the chain.

If possible, use a carbon copy, multiple page chain of custody form in order to maintain copies of the form throughout the shipment stages. If a multiple copy form is not used, photocopies should be made by the Stone personnel who originally ship the samples in order to document shipment.

4.2 Chain of Custody Procedures

1. The samples must be kept in the possession of the sampler until they can be securely packaged. The sampler should seal samples in a sturdy shipping box or other suitable container.
2. Fill out the COC form with all of the information listed in Section 4.1 above, except for items 9 and 10.
3. Attach the COC form to the outside of the shipping container such that the form can be filled out and signed each time the samples change possession or just inside the cooler. Alternately,

if the items transferred will only be relinquished once or shipped overnight via a courier service, the COC should be included within the shipping container(s). If applicable, a note may be made on the outside of the container to indicate the COC record is inside the container(s).

4. When the sampler delivers the sample container to the laboratory or to a courier, the sampler must enter on the COC form the date and time at which she/he relinquishes the container, then sign the form to signify that she/he has relinquished the container. The recipient of the container must then sign the form to signify that she/he has received the container, and she/he must also enter the date and time on the form.

5. This procedure must be followed each time the sample container changes hands. The first person must enter the date and time of relinquishment and sign the form, and then the next person must enter the date and time of receipt and sign the form. At each change of hands, the person relinquishing the container may take a copy of the form for his records, but the original form must remain with the container.

6. The exception to this procedure is that if a commercial courier is used (for example United Parcel Service, FedX, Airborne, etc.), the Stone tracking report, the bill of lading and/or tracking number from the courier will serve as valid COC documentation. **The courier need not sign the COC form to signify receipt of the samples.**

4.3 Laboratory Sample Receipt and Storage

When the laboratory receives the samples, the appropriate lab personnel should note the condition of the shipping container as described in the Laboratory Quality Systems Manual, then sign the COC form and enter the date and time the container is received. If applicable, the temperature of the sample container upon receipt will be recorded on the COC record. The completed, original form or a verified copy of the original should then be returned to Stone to be filed as a record of the sample possession history.

4.4 Documentation

If a multiple page carbon copy form is utilized copies should be maintained as follows:

- When the COC is filled out and ready to be placed in the shipping container, the sampler signs the COC and takes the bottom copy as a record that the samples were shipped.
- After receipt of samples by the testing facility or laboratory, the receiver signs the COC and may retain a copy (second page) of the COC for their records.
- The testing facility / laboratory should then forward the original COC (first page) to Stone for retention.
- Instructions for third party actions will be described on the COC form.

5.0 RESPONSIBILITIES

5.1 Quality Assurance Unit

1. If necessary and appropriate COC records may be audited by the quality assurance unit.

5.2 Principals/Group Officers/Group Leaders/Personnel

1. To maintain a record of sample collection, transfer between personnel, shipment, and receipt by the laboratory, a COC record will be completed for each sample shipment by the field sampler or field team.
2. No employee of Stone will collect or generate data, or perform any other study functions until they have been properly trained and such training is documented in a training record file as specified in SOP SEI-1.4.*n*.responsibilities.
3. It is the responsibility of the individual employees to follow SOPs covering activities in his/her work area or to identify a deviation from the written SOPs.

5.3 Archivist

The archivist is responsible for the following areas: maintaining of the original documents file including archival of all original data and archiving all original data as described in the SOP.

6.0 DEFINITIONS

1. *Raw data* any laboratory worksheets, records, memoranda, notes, or exact copies thereof, that are the result of original observations and activities of a study and are necessary for the reconstruction and evaluation of the report of that study. In the event that exact transcripts of raw data have been prepared (e.g., tapes which have been transcribed verbatim, dated, and verified accurate by signature), the exact copy or exact transcript may be substituted for the original source as raw data. Raw data may include photographs, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments.

7.0 REFERENCES

40 CFR Part 160 Good Laboratory Practice Standards, August, 1989.

8.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

1. Attachment 1: Sample Stone Chain of Custody Form

9.0 AUTHORIZATION

Reviewed by: John Hanzas Date: 7/5/2012

John Hanzas, Study Director/Senior GeoScientist

Approved by: Date: 6-28-12

Christopher T. Stone, President

10.0 REVISION HISTORY

Revision number 2:

1. Reformatted under new numbering scheme.

Revision number 3:

1. Section 3.0 (e) - added statement to clarify carrier's role in sample custody.
2. Added sample COC form.

Revision number 4:

1. Reformatted
2. Minor wording edits
3. Samplers may sign or initial form (section 2.1)

Revision number 5:

1. Reformatted.
2. Minor wording edits.
3. Add sections 2.2 and 3.7 detailing the use of multiple page COC forms.
4. Delete requirement of signature / initials of samplers (require only name(s)).
5. Allow for COC to be inserted inside container in section 3.3.

Revision number 6:

1. Change title to COC Procedures from COC Records.
2. Minor wording edits in Section 1.0, Objective.
3. Updated style to match SEI Style Guide. Reformatted using MS Word.
4. Added standardized section headers: 2.0 Policies, 3.0 Safety, 5.0 Responsibilities, 6.0 Definitions, 7.0 References, 8.0 Tables, Diagrams, Flowcharts and Validation data. Authorization moved to Section 9.0, and section10.0 Revision History.

Revision number 7:

1. Update Section 4.2 to allow for the COC to be inside the shipping container.
2. Change Federal Express to FedX

ATTACHMENT 1

Sample Stone Chain of Custody Form

CHAIN OF CUSTODY RECORD

STONE ENVIRONMENTAL INC

SEI Study # | Project Name/Client Study #

Study Director | Sampling Personnel (name/signature)

SAMPLE INFORMATION

Sample Identification	Date Collected	Type *	Cont.**	Number of Containers	Analyses Requested				
									Ship To:

Relinquished By: (Signature)	Date/Time	Received By: (Signature)	Date/Time
Relinquished By: (Signature)	Date/Time	Received By: (Signature)	Date/Time
Relinquished By: (Signature)	Date/Time	Received By: (Signature)	Date/Time

Special instructions:
* Type of sample (1) water (2) soil
** Container (1) bag (2) bottle (3) shelby tube (4) acetate sleeves (5) other: _____

Samples shipped with:
☐ ambient air ☐ dry ice ☐ ice

Temperature of samples when received by lab:
☐ room temperature ☐ cold ☐ frozen

Please remit completed, white copy to:

Stone Environmental, Inc.
535 Stone Cutters Way
Montpelier, Vermont 05602

Page __of __

white copy: original yellow copy: laboratory pink copy: SEI

I:\forms\custody\chain7.cdr

STONE ENVIRONMENTAL INC

Project Number:	Site Name/Location:	Samplers (Signature(s)):	Results and Invoice to: Stone Environmental, Inc. 535 Stone Cutters Way Montpelier, VT 05602
Client Name and Address:			Telephone #: 802-229-4541 Fax: 802-229-5417
Client Contact:		ANALYSES REQUESTED Container type[2]	Project Manager: PM Direct Phone #

Sample Identification	DATE/TIME Sampled		Matrix[1]	No. of Containers: Composite	No. of Containers: Grab									Remarks	Perservative added and final pH, if known[3]	Internal Billing Subproject Phase

Sampling Bottles/ Preservative Requirements

[1]Matrix: SL-Soil, SG-Soil Gas, GW-Groundwater, SD-Sediment, SW-Surface Water, WW-wastewater, L-liquid, Wipe, Solid, Other-specify_____(i.e., oil, concrete)

[2]Container(s): V-VOA 40mL vials, T-Tedlar Airbag, A - Amber 1L, G-Glass, 1L, M-250 mL-Glass Wide Mouth, P-poly/plastic, O-other, specify_____

Metals, check one: ___8RCRA, ___13 PP, ___23 TAL, ___ 14MCP. Method: ___6010, ___ 6020, ___200.7 Other:_____

Dissolved Metals Field Filtered? ___Yes ___No

[3]Preservative: HCL, MeOH, HNO3, H2SO4, NaOH, O-Other, Specify_____

Samples shipped: _____ice, _____dry ice, _____ambient

Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Date/Time	Shipped Via: Number:
Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Date/Time	Turnaround Time Requirements: <14days must have Pre-Authorization.
Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Date/Time	

Notes/Comments: Custody Seal Intact?: Y N N/A Sample Temp at Lab.

EDD Required: Specify Type?_____, (i.e., Equis EZLoad, Excel spreadsheet, Other)

Report Package level needed: ___Level II (results, SS recoveries), ___ Level III (results, all QC), ____Level IV (FULL, CLP-like)

STONE ENVIRONMENTAL INC
CHAIN-OF-CUSTODY RECORD

Page _____ of _____

white copy: original yellow copy: laboratory pink copy: Stone

Revised 8/4/2011
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STANDARD OPERATING PROCEDURE

SEI-4.5.11

DATA HANDLING, STORAGE, RETRIEVAL AND ERROR CODING

SOP Number: SEI-4.5.11
Revision Number: 11

Date Issued: 09/02/93
Date of Revision: 05/10/12

1.0 OBJECTIVE

This standard operating procedure (SOP) describes the framework for handling study specific (or project specific) raw data and documentation obtained during the process of a study. Provided in this SOP are the essential elements necessary to accurately record activities, measurements, and observations made in the field or during the course of a study and during the processing of data that ultimately is used in a project report.

Definition of Raw Data: *Raw data* any laboratory worksheets, records, memoranda, notes, or exact copies thereof, that are the result of original observations and activities of a study and are necessary for the reconstruction and evaluation of the report of that study. In the event that exact transcripts of raw data have been prepared (e.g., tapes which have been transcribed verbatim, dated, and verified accurate by signature), the exact copy or exact transcript may be substituted for the original source as raw data. Raw data may include photographs, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments.

2.0 POLICIES

1. Personnel will legibly record data and observations in the field to enable others to reconstruct project events and provide sufficient evidence of activities conducted.
2. All data generated during the conduct of a study, except those that are generated by automated data collection systems, shall be recorded directly, promptly, and legibly in indelible ink.
3. All data entries shall be dated on the day of entry and signed or initialed by the person entering the data. If multiple personnel enter data upon a single page, each entry should be uniquely attributed to its author.
4. Field personnel will use a field logbook, observation and remark form or other designated field form to record activities, measurements, and observations made in the field.

5. Any change in entries shall be made so as not to obscure the original entry, shall indicate the reason for such change and shall be dated and signed or identified at the time of the change. Procedures to follow for error coding are discussed in the procedural section of this SOP.
6. Personnel will document the procedures performed in accordance with Good Laboratory Practices (GLP) Standards, 40 CFR Part 160.
7. All data will be stored in project specific files. All raw data will be archived in accordance with SOP SEI-7.2.*n*.
8. Any access into the archives must be documented in the "Archive Access Log", located with the archivist.

3.0 SAFETY ISSUES

Not applicable to this SOP

4.0 PROCEDURES

Determine the required data to be collected. This can be ascertained from several sources, including: (1) an approved protocol, (2) an approved work plan or Quality Assurance Project Plan (QAPP), (3) an awarded bid, (4) or other documentation that sets forth study design agreed upon by the client and Stone.

4.1 Use and Assembly of Field Study Notebooks and Logbooks

Prior to initiating fieldwork, to the extent possible, a field study notebook or logbook should be obtained and/or assembled to provide an accurate and permanent record of activities, measurements, and observation made in the field. The field notebook should be a self-contained binder that will assist in protecting the field forms, or other appropriate recording media from damage or loss. Once the type of data to be collected is known, search of the Forms subdirectory on Stone's network for specific task related forms which can be customized for a project-specific study or project. Blank forms should be created to be study-specific and saved within the project directory on the Stone network in such a manner that it is clear where the forms are located so any study personnel can print forms as needed for project work.

As forms are saved to the project directory, type in all data that may include, but is not limited to:

1. Stone study number or project number
2. Sponsor or client identification number (if available)
3. Project Location
4. Study Director or Project Manager
5. Study Title
6. Wherever practical include event number, target date and sample identification (ID)

Print copies of all forms as needed. Retain originals and insert a master copy into the notebook. The master copy is then available for photocopying should you run out of forms in the field.

The binder should be labeled in an appropriate manner to aid in identification of the field study notebook.

An approved protocol, work plan or reference QAPP should be included in the notebook. Other items to be included in the notebook, may consist of the following:

1. Applicable protocol or method modifications and deviations as necessary and appropriate.
2. Approved Site Health and Safety Plan (only if it cannot be stored on site).
3. SOPs for data collection.

All original loose-leaf field notes shall be submitted for inclusion in the study or project records.

4.2 Field Data Collection and Handling

All field notes and records must be accurate, orderly, complete, and legible.

A pen (preferably black) or an indelible marker should be used for note taking. Label each page with the date, the signature of the person taking notes (initials may also be appropriate), and the page number. All notes, signatures, and other observations should be entered in the field at the time the notes are taken.

If multiple field personnel make entries onto any sheet or form, it must be clear who entered what data via initialing, bracketing, or other methods.

Any changes in entries shall be made so as not to obscure the original entry, shall indicate the reason for such change, and shall be dated and signed or initialed at the time of the change.

A legible record of data and observations recorded in the field should be sufficient to enable others to reconstruct field events and provide sufficient evidence of activities conducted.

Forms may be removed during actual data collection, but should then be placed in a suitable protective booklet or folder (e.g. a metal field notepad). Every effort should be made to protect data sheets from physical elements that may cause the data to become illegible or lost. Data sheets should be returned to the notebooks as soon as possible (i.e. by the end of the day).

Data may also be recorded on the pre-printed Stone Observation and Remarks Form (O&R) and then included in the project files or the field study notebook.

4.2.1 Error Coding

When errors to the raw data are made or discovered, the original entry should be crossed through with one line in a manner as to not obscure the original entry and designated with a

footnote number. The footnote number should be circled in order to separate it from any surrounding data. The correction should be initialed, dated, and justified in a footnote at the bottom of the page or at the point of entry. The code justification or reason for the edit should be circled to limit confusing the code with the editor's initials. Each entry correction must be given an individual footnote.

Each entry correction must be given an individual footnote number and justified, initialed, and dated at the bottom of the page or on the appropriate medium (see example 1). The following data correction symbols may be used in justifying corrections:

C - Calculation Error	TE – Transcription Error
E - Entry Error	LE- Late Entry
S - Spelling Error	F - Form Change
R - Rounding Error	W - Write Over

(1)

September 15, ~~1997~~1998

(1)(E) RWP, 9/15/98 (1)

[(1)(2)(3)] (LE)(E) RWP, 9/15/98 (2)

The person who originally entered the data should make error corrections. If the person who entered the original data is unavailable, the edit must clearly be supported with appropriate documentation and justification for corrections as noted above.

Any correction made at a later date than the original entry **must** be followed by an "LE" (late entry) designation. This must be footnoted and explained at the bottom of the original data form, on an observation and remarks form, or another suitable medium to clarify the reason for the change and/or addition to the original entry. Similar Late Entry changes may be combined with one set of initials and date, but must still be issued with individual footnote numbers and all footnotes must be contained within one set of brackets (see example (2) above).

Please note that if data generated during the conduct of a study are not recorded directly, promptly, and legibly in ink and all data entries were not dated on the day of entry and signed or initialed by the person entering the data, then "Late Entry" corrections must be indicated in the GLP compliance statement. For example, "The activities which occurred prior to October 12, 2000 were not documented directly and promptly. This was a violation of 40 CFR Part 160.130(e). It is not anticipated that these violations will have an adverse impact on the study."

If multiple errors **of the same type** are discovered at the same time (e.g. during review of page prior to sign off), these errors and corrections can be documented similar to LE changes as above (see example 2). *Note: every effort should be made to make corrections/changes to data*

entries at the time of their discovery. DO NOT wait until other data is recorded and then go back and make corrections.

It some cases, in the on-site laboratory when corrections are made only the initials and date of the corrections are necessary since these corrections are always typically documented as an "entry error" and this type of correction is acceptable according to the National Environmental Laboratory Standards.

4.3 Automated Field Data Collection

In automated data collection systems, the individual responsible for direct data input shall be identified at the time of data input. Any change in automated data entries shall be made so as not to obscure the original entry, shall indicate the reason for change, shall be dated, and the responsible individual shall be identified.

4.4 Certification of Exact Copies of Data

"Raw data" means any laboratory worksheets, records, memoranda, notes, or exact copies thereof, that are the result of original observations and activities of a nonclinical laboratory study and are necessary for reconstruction and evaluation of the report of that study. **In the event that exact transcripts of raw data have been prepared (e.g., tapes which have been transcribed verbatim, dated, and verified accurate by signature), the exact copy or exact transcript may be substituted for the original source as raw data.** When raw data is copied, the copy will be stamped with the following:

"This is an exact copy of the original document."

By_____ Date_____

In the event that there are multiple copies required (e.g., weather data for multiple projects) then the top page should be stamped as above and (if available) the number of pages noted on the top page. At the end of a study all raw data is copied and shipped to the sponsor in accordance with SOP SEI-7.1.*n*.

Copies of transcripts or documents that are not considered raw data are not required to be stamped as above. However, if in doubt, all copies should be stamped as noted above unless otherwise noted.

4.5 Storage of Data

Data will be stored in specific project files. Original raw data sheets will be stored in accordance with SOP SEI-7.2.*n*. All raw data, documentation, records, protocols, specimens, and final reports generated as a result of a study shall be retained. Correspondence and other documents relating to interpretation and evaluation of data, other than those documents contained in the final report also shall be retained as described in 40 CFR 160 § 160.190.

Materials retained or referred to in the archives shall be scanned electronically to permit expedient retrieval.

All study specific computer documentation should ultimately be stored on the network hard drive. Back-up copies will be made of all hard drives on a set schedule as described in the SOP SEI-1.6.n.

4.6 Retrieval of Data

All project data, including both active and inactive project data, will be kept in Stone's filing room, which is kept locked at all times. Archived project data shall be kept separate from active project data in locked filing cabinets.

Any active project data may be accessed by staff if accompanied by the archivist. Any access into the archives must be documented in the "Archive Access Log" located with the archivist (see SOP SEI-7.2.*n*).

Appropriate original raw data, protocols, and final reports shall be located in the archives. Authorized personnel may only access archived data.

5.0 RESPONSIBILITIES

1. All personnel will legibly record data and observations (including phone conversations) in accordance with this SOP to enable others to reconstruct project events and provide sufficient evidence of activities conducted.

2. All personnel will label each page with the date, the signature of the person taking notes (initials may also be appropriate), and the page number. All notes, signatures, and other observations should be entered in the field at the time the notes are taken.

3. If multiple personnel make entries onto any sheet or form, it must be clear who entered what data via initialing, bracketing, or other methods.

6.0 DEFINITIONS

1. *Archives* Include an area for orderly storage and expedient retrieval of original raw data, documentation, protocols, specimens, and interim and final reports retained as a result of a study. Conditions of storage shall minimize deterioration of the documents or specimens in accordance with the requirements for the time period of their retention and the nature of the documents of specimens. An individual shall be identified as responsible for the archives. Only authorized personnel shall enter the archives. Material retained or referred to in the archives shall be indexed to permit expedient retrieval. A testing facility may contract with commercial archives to provide a repository for all material to be retained. Raw data and specimens may be retained elsewhere provided that the archives have specific references to those other locations.

2. *EPA* the U.S. Environmental Protection Agency.

3. *FIFRA* the Federal Insecticide, Fungicide, and Rodenticide Act as amended.

4. *Observations & Remarks Form (O&R):* A pre-printed form, which contains mostly blank space for general note taking. The form typically prompts the user for the study or project designation, the SEI project number, the client or sponsor name, the total number of pages (page *n* of n) and requires a signature and date. The form is generally used to capture notes of one person when another, more specific forms is not available.

5. *Quality Assurance Unit (QAU)* any person or organizational element except the study director, designated by testing facility management to perform the duties related to quality assurance of the studies.

6. *Raw data* any laboratory worksheets, records, memoranda, notes, or exact copies thereof, that are the result of original observations and activities of a study and are necessary for the reconstruction and evaluation of the report of that study. In the event that exact transcripts of raw data have been prepared (e.g., tapes which have been transcribed verbatim, dated, and verified accurate by signature), the exact copy or exact transcript may be substituted for the original source as raw data. Raw data may include photographs, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments.

7. *Study* means any experiment at one or more test sites, in which a test substance is studied in a test system under laboratory conditions or in the environment to determine or help predict its effects, metabolism, product performance (pesticide efficacy studies only as required by 40 CFR 158.640) environmental and chemical fate, persistence, or residue, or other characteristics in humans, other living organisms, or media. The term "study" does not include basic exploratory studies carried out to determine whether a test substance or a test method has any potential utility.

7.0 REFERENCES

40 CFR Part 160 Good Laboratory Practice Standards, August, 1989.

8.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

None

9.0 AUTHORIZATION

Reviewed by: _____ Date: 7/5/2012

John Hanzas, Study Director/Senior GeoScientist

Approved by: _____ Date: 6-27-12

Christopher T. Stone, President

10.0 REVISION HISTORY

Revision number 2 (SEI-4.3.2):

1. Reformatted under new numbering scheme.

Revision number 3 (SEI-4.3.3):

1. Reformatted
2. Minor wording edits.

Revision number 4 (SEI-4.3.4):

1. Reformatted
2. Minor wording edits
3. Add section 2.4 detailing documentation of entries by multiple personnel.

Revision number 5 (4.3.5)

1. Minor wording edits.

Retire SOP SEI-4.3.5, Combine with 4.5.9.

Revision number 1 (4.11.1):

1. Reformatted under new numbering scheme.

Revision number 2 (4.11.2):

1. Reformatted.
2. Minor wording edits.

Retire SOP SEI-4.11.2, Combine with 4.5.9.

Revision number 2 (4.5.2):

1. Reformatted under new numbering scheme.

Revision number 3 (4.5.3):

1. Inserted the requirement of using an "indelible" black ink pen in section 2.1 (2).
2. Added clarification that each entry changed must receive it own footnote number and explanation in section 2.1 (4).

Revision number 4 (4.5.4):

1. Deleted the requirement of using a black ink pen in section 2.1 (2).
2. Deleted EC (entry change) from the list of standard codes.
3. Deleted W (wording change) from the list of standard codes.
4. Added W for "write over".

Revision number 5 (4.5.5):

1. Section 2.1 part 4 - added circling of the footnote number **and** the error code when justifying an error/change in the raw data.

Revision number 6 (4.5.6):

1. Reformatted

Revision number 7 (4.5.7):

1. Reformatted

Revision number 8 (4.5.8):

1. Add detail to section 2.1.4 regarding who can make changes to data.

Revision number 9:

1. Title changed to Data Handling, Error Coding, Storage and Retrieval.
2. Incorporate SEI- SOPs; SEI-4.11.2, Assembly of Field Study Notebooks and SEI-4.3.5 Use of Field Logs into Data Handling SOP.
3. Minor wording edits in Section 1.0, Objective.
4. Updated style to match SEI Style Guide – font and text. Reformatted using MS Word.
5. Added standardized section headers: 2.0 Policies, 3.0 Safety, 5.0 Responsibilities, 6.0 Definitions, 7.0 References, 8.0 Tables, Diagrams, Flowcharts and Validation data. Authorization moved to Section 9.0, and section10.0 Revision History.

Revision number 10:

1. Added Section 4.4 on Copies. As a result, subsequent sections were re-numbered.

Revision number 11:

1. Updated wording to reflect Stone's current formatting.
2. Added "Materials retained or referred to in the archives shall be scanned electronically to permit expedient retrieval" so Section 4.5 Storage of Data.
3. Updated Section 4.6 Retrieval of Data to reflect Stone's new policy of having a locked filing room.

STANDARD OPERATING PROCEDURE

SEI-5.1.5

MAINTENANCE AND DECONTAMINATION OF FIELD EQUIPMENT

SOP Number: SEI-5.1.5
Revision Number: 5

Date Issued: 04/09/93
Date of Revision: 02/20/04

1.0 OBJECTIVE

Proper maintenance and decontamination of field equipment is an essential quality control measure and a safety precaution to ensure sampling equipment is in proper working condition and to remove any chemical or material contamination. The procedure prevents cross-contamination between sampling stations and helps maintain a clean working environment for the safety of all field personnel. Equipment decontamination helps prevent sampling bias. Maintenance records of equipment that "generates, measures, or assesses data" is required under the Good Laboratory Practices (GLPs) and is an important element of a compliant and scientifically sound operation. This standard operating procedure (SOP) defines the difference between maintenance of equipment and general decontamination procedures and sets forth the guideline SEI uses in determining if equipment records are required per Subpart D of 40 CFR Part 160. This SOP describes a general procedure for decontaminating a sampling device, more specific maintenance requirements and decontamination procedures may be described in SOPs specific to each type of equipment.

2.0 POLICIES

1. According to 40 CFR Part 160, Subpart D, Section 160.61, Equipment used in the generation, measurement, or assessment of data and equipment used for facility environmental control shall be of appropriate design and adequate capacity to function according to the protocol and shall be suitable located for operation, inspection, cleaning, and maintenance.
2. Personnel will legibly record data and observations in the field to enable others to reconstruct project events and provide sufficient evidence of activities conducted.
3. All data generated during the conduct of a study, except those that are generated by automated data collection systems, shall be recorded directly, promptly, and legibly in indelible ink.
4. All data entries shall be dated on the day of entry and signed or initialed by the person entering the data. If multiple personnel enter data upon a single page, each entry should be uniquely attributed to its author.
5. Personnel will document the procedures performed in accordance with Good Laboratory Practices (GLP) Standards, 40 CFR Part 160.

3.0 SAFETY ISSUES

1. If necessary and appropriate, a site-specific health and safety plan shall be created for each study site. A template for creating a proper health and safety plan is provided on the SEI network.
2. If necessary and appropriate, all chemicals are required to be received with a Material Safety Data Sheets (MSDS) or appropriate application label. These labels or MSDS shall be made available to all personnel involved in the sampling and testing.

4.0 PROCEDURES

4.1 Equipment types And Maintenance and Calibration Requirements

The majority of SEI's field equipment does not directly generate, measure or assess data and thus does not require the full documentation as described in Subpart D of the GLPs regarding written record keeping of all inspections, maintenance, testing, calibration or standardization operations.

Examples of exempt equipment include, but are not limited to:

1. Hand augers
2. Pump controllers/compressors
3. Generators
4. General hand tools (shovels, hammers, etc.)
5. Well pumps (bladder, electric submersible, etc)
6. Sampling Tools (bailers, dipping tools)

Maintenance documentation may be kept for this type of equipment to assist with inventory or the general condition of the equipment, but the records may not include all items required under 40 CFR Part 160, Subpart D.

Examples of equipment which fall under 40 CFR Part 160, Subpart D include, but are not limited to:

1. pH, conductivity meters.
2. Water level indicators.
3. Troll datalogger (for slug tests, etc.)
4. Guelph permeameter.
5. Scales (balances)

6. Flow meters.

7. Gas Chromatographs

For equipment, which falls under 40 CFR Part 160, Subpart D, at a minimum, the following records must be maintained:

1. An SOP must be written which details the methods, materials and schedules for the routine inspection, cleaning, maintenance, testing, and calibration for the equipment. Details will also include remedial action that will be taken in the event of failure or malfunction of the equipment. The SOP will include (by title if appropriate) the person responsible for maintenance of the equipment (both routine and non-routine).

2. Records will be maintained of all inspection, maintenance, testing, and calibrating operations. These records will contain, at a minimum:

    a. Dates of operation.

    b. Whether maintenance was routine or non-routine.

    c. Whether maintenance followed written SOPs.

3. If non-routine repairs are performed, at a minimum, the following will be documented in addition to the items in 2 above:

    a. Nature of the defect.

    b. How and when the defect was discovered.

    c. Any remedial or correction action taken in response to the defect.

Inspection, maintenance, testing and calibration efforts will be documented. In general, a checklist form or a maintenance logbook, specifically developed for each piece of equipment, should be used to document these procedures. This form or logbook should prompt the user for the maintenance, etc. to be performed. If a form or logbook is not available, actions must still be documented on an appropriate medium. Forms for equipment must be taken to the field for use. Once field efforts are complete, the forms should be returned to SEI's main office for filing. All original equipment maintenance forms will be retained in a filing cabinet. Copies may be made for inclusion in data packages at the request of a client. Records shall be maintained on each major item of equipment and all reference materials significant to the test performed. As indicated above these records shall include documentation on all routine and non-routinge maintenance activities and reference material verification. If available, the records shall include:

1. the name of the item of equipment;

2. The manufacturer's name, type identification, and serial number or other unique identification;

3. Date received and date place in service (if available);

4. Current location; where appropriate;

5. Condition when received (e.g., new, used, reconditioned);

6. Copy of the manufacturer's instructions,

7. Dates and results of calibrations and/or verifications and date of the next calibration and/or verification;

8. Details of maintenance carried out to date and planned for the future; and,

9. History of any damage, malfunction, modification or repair

10. Equipment which is malfunctioning and has been taken out of service, it shall be tagged as such.

If a piece of equipment is dedicated to a sampling site/study, the maintenance forms or logbook may be retained with that equipment until complete or the fieldwork is finished. Once either of these criteria has been fulfilled, the form should be filed in the project file or the general equipment file as noted above.

4.2 Support Equipment

The following are the calibration and maintenance requirements for laboratory analytical support equipment. This applies to all devices that may not be actual test instrument, but are necessary to support laboratory operations. These include but are not limited to: balances, ovens, refrigerators, freezers, incubators, water baths, temperature measuring devices (including thermometers and thermistors, thermal/pressure sample preparation devices and volumetric dispensing devices (such as Eppendorf®, or automatic dilutor/dispensing devices) if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume

1. All support equipment shall be maintained in proper working order. The records of all repair and maintenance activities including service calls shall be kept.

2. All support equipment shall be calibrated or verified at least annually, using NIST traceable references when available, over the entire range of use. The results of such calibration shall be within the specifications required of the application for which this equipment is used or:

    a. The equipment shall be removed from service until repaired; or

b. The laboratory shall maintain records of established correction factors to correct all measurements

3. Raw data records shall be retained to document equipment performance.

4. Prior to use on each working day, balances, ovens, refrigerators, freezers, and water baths shall be checked in the expected use range, with NIST traceable references where available. The acceptability for use or continued use shall be according to the needs of the analysis or application for which the equipment is being used.

5. Mechanical volumetric dispensing devices including burettes (except Class A glassware) shall be checked for accuracy on at least a quarterly use bases. Glass micro liter syringes are to be considered in the same manner as Class A glassware, but must come with a certificate attesting to established accuracy or the accuracy must be initially demonstrated and documented by the laboratory

6. For chemical tests, the temperature, cycle time, and pressure of each run auto claves must be documented by the use of appropriate chemical indicators or temperature recorders and pressure gauges.

4.3 Equipment Decontamination and Personnel

Decontamination of equipment does not constitute maintenance, but is a specific step required for the use of equipment between sampling points. Decontamination is a requirement for use of the equipment but does not effect whether the equipment is in working order. Improper decontamination may effect the quality or integrity of a sample, but does not effect the device collecting the sample. Thus, decontamination is not an action, which must be documented per 40 CFR Part 160, Subpart D.

Decontamination of sampling equipment refers to the physical and chemical steps taken to remove any chemical or material contamination. Equipment decontamination helps prevent sampling bias. All equipment that comes in contact with the sampled material should be free of components that could influence (contaminate) the true physical or chemical composition of the material. Besides the equipment used to collect the samples, any containers or equipment used for sample compositing or for field sub sampling should be free of contamination.

Equipment decontamination also prevents cross-contamination of samples when the equipment is used to collect more than one sample. Disposable equipment or the use of dedicated equipment provides the most effective means of avoiding cross-contamination; however, the use of such equipment is not always practical.

You should decontaminate equipment to a level that meets the minimum requirements for your data collection effort. Your decontamination steps (e.g., use of solvents versus use of only soap and water), therefore, should be selected based on the constituents present, their concentration levels in the waste

or material sampled, and their potential to introduce bias in the sample analysis results if not removed from the sampling equipment. You should describe the project specific decontamination procedures in your planning document for the sampling effort. In addition, items used to clean the equipment, such as bottlebrushes, should e free of contamination.

The following procedures is an example of one you could use to decontaminate sampling device to be used for collecting samples for trace organic or inorganic constituent analyses (from USEPA 1996):

1. Clean the device with tap water and soap, using a brush if necessary to remove particulate matter and surface films.

2. Rinse thoroughly with tap water.

3. Rinse thoroughly with analyte- or organic-free water.

4. If necessary and appropriate, rinse thoroughly with solvent. Do not solvent-rinse PVC or plastic items.

5. Rinse thoroughly with organic/analyte free water, or allow equipment to dry completely.

6. Remove the equipment from the decontamination area. Equipment stored overnight should be wrapped in aluminum foil and covered with clean, unused plastic.

The specifications for cleaning materials are as follows (you should justify and document the use of substitutes):

- "Soap" should be phosphate-free laboratory detergent such as Liquinox®. It must be kept in a labeled, clean, plastic, metal, or glass containers until used and poured directly from the container when in use.

- "Solvent" should be pesticide-grade isopropanol. It must be stored in the unopened original containers until used. It may be applied using the low-pressure nitrogen system fitted with Teflon nozzle, or using Teflon® squeeze bottles. For equipment highly contaminated with organics (such as oily wastes), a laboratory-grade hexane may be more suitable alternative to isopropanol or for volatile organic contaminates purge and trap grade methanol may be more appropriate.

- "Tap Water" may be used from any municipal water treatment system. Use of an untreated potable water supply is not an acceptable substitute unless the source has been determined to be free of contaminates of interest. Tap water may be kept in clean tanks, hand pressure sprayers, squeeze bottles, or applied directly form a hose of tap.

- "Analyte free water (deionized water) is tap water treated by passing through a standard deionizing resin column. At a minimum, it must contain no detectable heavy metals or other inorganic compounds as defined a standard ICP (or equivalent scan. It may be obtained by other methods as long as it meets the analytical criteria. Analyte free water must be stored in clean glass, stainless steel, or plastic containers that can be closed prior to use. IT can be applied from plastic squeeze bottles.

- "Organic/analyte free water" is tap water that has been treated with activated carbon and deionizing units. A potable system to produce such water under field conditions is available. At a minimum, the water must meet the criteria of analyte free water and not contain detectable pesticides, herbicides, or extractable organic compounds, and no volatile organic compounds above minimum detectable levels as determined for a given set of analyses. Organic/analyte free water obtained by other methods is acceptable, as long as it meets the analytical criteria. It may be applied using Teflon® squeeze bottles or with the portable system.

Clean the field equipment prior to field use. Designate a decontamination zone at the site and, if necessary, construct a decontamination pad at a location free of surface contamination. If necessary, you should collect wastewater from decontamination (e.g., via a sump or pit) and remove it frequently for appropriate treatment or disposal. The pad or area should not leak contaminated water into the surrounding environment. You also should collect solvent rinses for proper disposal.

You should always handle field-cleaned equipment in a manner that prevents recontamination. For example, after decontamination but prior to use store, the equipment in a location away from the cleaning area and in an area free of contaminants. If it is not immediately reused, you should cover it with plastic or aluminum foil to prevent recontamination.

At certain sites, decontamination will generate a quantity of wastes called investigation derived waste (IDW). You should address the handling and disposal of IDW in your sampling plan. You must handle this material in accordance with whether it is nonhazardous or suspect of, or know to be, hazardous. You should minimize the generation of hazardous IDW and keep it separated from the nonharzardous IDW. For example, you should control the volume of spent solvents during equipment decontamination by applying the minimum amount of liquid necessary and capturing it separately form the nonhazardous wash water. For additional guidance on handling IDW, see Management of Investigation-Derived Wastes (USEPA 1992).

Decontamination of personnel and their protective gear also is often necessary during hazardous waste sampling. This important type of decontamination protects personnel from chemical exposure and prevents cross-contamination when personnel change locations. The level or degree of such decontamination will depend on site-specific considerations, such as the decontamination procedures in your health and safety plan. For additional information regarding decontamination, See ASTM D5088, Standard Practice for Decontamination of Field Equipment Used at Nonradioactive Waste

Sites. Another source of additional information is "Sampling Equipment Decontamination (USEPA 1994) issued by EPA's Environmental Response Team.

If necessary and appropriate equipment specific decontamination procedures will be outlined in equipment specific SOPs. These SOPs will clearly state the type of documentation required for each type of equipment and state whether the equipment falls under 40 CFR part 160, subpart D. Decontamination may be documented by citing an SOP or directly on a sampling form or other task specific medium.

5.0 RESPONSIBILITIES

1. All personnel will legibly record data and observations (including phone conversations) in accordance with this SOP to enable others to reconstruct project events and provide sufficient evidence of activities conducted.

2. Prior to use and after use, all equipment will be appropriately cleaned and decontaminated in accordance to this SOP.

6.0 DEFINITIONS

1. *Archives* - Include an area for orderly storage and expedient retrieval of original raw data, documentation, protocols, specimens, and interim and final reports retained as a result of a study. Conditions of storage shall minimize deterioration of the documents or specimens in accordance with the requirements for the time period of their retention and the nature of the documents of specimens. An individual shall be identified as responsible for the archives. Only authorized personnel shall enter the archives. Material retained or referred to in the archives shall be indexed to permit expedient retrieval. A testing facility may contract with commercial archives to provide a repository for all material to be retained. Raw data and specimens may be retained elsewhere provided that the archives have specific references to those other locations.
2. *Calibration* – To determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter, instrument, or other device. The levels of the applied calibration standard should bracket the range of planned or expected sample measurement (NELAC).
3. *Decontamination* – Procedures followed to ensure cross contamination does not occur between sampling points or that potential contamination of equipment does not pose a hazard to sampling personnel.
4. *EPA* the U.S. Environmental Protection Agency.
5. *Equipment which Generates Data* – Equipment which directly collects a data point (or multiple data points) necessary for study goals and stores or displays the data for viewing.
6. *Equipment which Measures Data* – Equipment which directly collects a data point (or multiple data points) which includes a unit that can be used to compare the data point (or points which includes a unit that can be used to compare the data point (or points) with other data of the same unit.

7. *Equipment which Assesses Data* - Equipment which collects a data point (or multiple points) and manipulates the data in a manner in which inferences or calculations are made which create data other then what was directly collected. Or, equipment which measures the performance or accuracy of the data it collects.

8. *FIFRA* the Federal Insecticide, Fungicide, and Rodenticide Act as amended.

9. *Maintenance* – Actions performed on equipment to standardize and/or correct the accuracy and precision of a piece of equipment to ensure that the equipment is operating within the manufacturer's specifications and standard values.

10. *Observations & Remarks Form (O&R):* A pre-printed form, which contains mostly blank space for general note taking. The form typically prompts the user for the study or project designation, the SEI project number, the client or sponsor name, the total number of pages (page *n* of n) and requires a signature and date. The form is generally used to capture notes of one person when another, more specific forms is not available.

11. *Quality Assurance Unit (QAU)* any person or organizational element except the study director, designated by testing facility management to perform the duties related to quality assurance of the studies.

12. *Raw data* any laboratory worksheets, records, memoranda, notes, or exact copies thereof, that are the result of original observations and activities of a study and are necessary for the reconstruction and evaluation of the report of that study. In the event that exact transcripts of raw data have been prepared (e.g., tapes which have been transcribed verbatim, dated, and verified accurate by signature), the exact copy or exact transcript may be substituted for the original source as raw data. Raw data may include photographs, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments.

13. *Study* means any experiment at one or more test sites, in which a test substance is studied in a test system under laboratory conditions or in the environment to determine or help predict its effects, metabolism, product performance (pesticide efficacy studies only as required by 40 CFR 158.640) environmental and chemical fate, persistence, or residue, or other characteristics in humans, other living organisms, or media. The term "study" does not include basic exploratory studies carried out to determine whether a test substance or a test method has any potential utility.

7.0 REFERENCES

40 CFR Part 160 Good Laboratory Practice Standards, August, 1989.

USEPA. 1992. Management of Investigation-Derived Wastes. Directive 9345.3-03FS. NTIS PB92-963353. Office of Solid Waste and Emergency Response. Washington, DC.

USEPA . 1994. "Sampling Equipment Decontamination." Environmental Response Team SOP#2006, Revision#0.0. Edison, NJ. http://www.ert.org/

USEPA. 2000. National Environmental Laboratory Accreditation Conference (NELAC), EPA 600/R-00-0084Office of Research and Development, Las Vegas, NV.

USEPA. August 2002. RCRA Waste Sampling Draft Technical Guidance, EPA 530-D02-002, Office of Solid Waste and Emergency Response. Washington, DC. (5305W)

8.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

None

9.0 AUTHORIZATION

Revised by: Kim B. Watson Date: 2-19-04

Kim B. Watson, Quality Assurance Manager

Approved by: Christopher T. Stone Date: 2-19-04

Christopher T. Stone, President

10.0 REVISION HISTORY

Revision number 1

1. Added Section 3.0 number 10, for storage requirements.
2. Reformatted under new numbering scheme.

Revision number 2:

1. Changed wording of introduction to state that decontamination is a quality *control*, not a quality assurance measure.

Revision number 3:

1. Reformatted
2. Minor wording edits
3. Remove air dry requirement (section 3, # 8)

Revision number 4:

1. Change title.
2. Re-formatted.
3. Added definitions.
4. Added detail of the type of documentation required under subpart D of the GLPs.
5. Added section 3.4 and 3.5 regarding the use of checklists and filing of equipment documentation.
6. Added detail of what decontamination constitutes.

Revision number 5:

1. Minor wording edits in Section 1.0, Objective.
2. Updated style to match SEI Style Guide – font and text. Reformatted using MS Word.
3. Added standardized section headers: 2.0 Policies, 3.0 Safety, 5.0 Responsibilities, 6.0 Definitions, 7.0 References, 8.0 Tables, Diagrams, Flowcharts and Validation data. Authorization moved to Section 9.0, andSection10.0 Revision History.
4. Revised Section 4.2 on Decontamination of Equipment and Personnel to include general decontamination procedures as outlined in the RCRA Waste Sampling Draft Technical Guidance Document (USEPA 2002).
5. Added Section on Support Equipment for the on-site laboratory.
6. Included Section under Section 4.1 on records requirements for equipment.
7. Updated References

STANDARD OPERATING PROCEDURE

SEI-5.26.0

USE, MAINTENANCE AND CALIBRATION OF THE LAMOTTE MODEL 2020e TURBIDITY METER

SOP Number: SEI-5.26.0
Revision Number: NA

Date Issued: 6/23/05
Date of Revision: NA

1.0 OBJECTIVE

This Standard Operating Procedure describes the use, maintenance and calibration of the LaMotte Model 2020e, turbidity meter. Turbidity is measured by detecting and quantifying the scattering of light in water. The 2020 is a portable microprocessor controlled, direct reading colorimeter and nephelometer that uses a tungsten filament light source that meets or exceeds EPA specifications and is designed for a uniform light spot image and stable output. The turbidity meter generates and measures data, and thus must meet the requirements of 40 CFR part 160 subpart D.

2.0 POLICIES

1. According to 40 CFR Part 160, Subpart D, Section 160.61, Equipment used in the generation, measurement, or assessment of data and equipment used for facility environmental control shall be of appropriate design and adequate capacity to function according to the protocol and shall be suitable located for operation, inspection, cleaning, and maintenance.
2. Personnel will legibly record data and observations in the field to enable others to reconstruct project events and provide sufficient evidence of activities conducted.

3.0 SAFETY ISSUES

1. If necessary and appropriate, a site-specific health and safety plan shall be created for each study site. A template for creating a proper health and safety plan is provided on the SEI network.
2. If necessary and appropriate, all chemicals are required to be received with a Material Safety Data Sheets (MSDS) or appropriate application label. These labels or MSDS shall be made available to all personnel involved in the sampling and testing.

4.0 PROCEDURES

4.1 Equipment and Materials

1. LaMotte Model 2020e Turbidity Meter and users manual
2. Appropriate turbidity standard solutions
3. Glass sample tubes
4. Positioning rings
5. Equipment calibration Log or other appropriate medium to record calibration data

Prior to use in the field the user should read and understand the manufacturer's operating instruction manual which is included in the carrying case and a copy is also provided in the equipment maintenance file.

4.2 Selecting a Calibration Curve Reference

1. The default units are NTU (nephelometric turbidity units) and the default calibration curve reference is formazin.

2. Turn the meter on and scroll down to **Options**, press* | **OK**.

3. Scroll down and then press * | **OK** to select **Turbidity**.

4. Scroll down and then press * | **OK** to select **Calibration**.

5. Scroll down and then press * | **OK** to select a **Calibration** curve (formazin). Select a calibration option based on the composition of the standards that will be used to calibrate the meter.

6. For the most accurate results, the Attenuation option should be chosen when samples are over 500 NTU. The range for the Attenuation option is 40-4000 NTU.

4.3 Calibration Procedure

Prior to use, a standard will be scanned. The reading will be verified. The meter will be recalibrated if necessary.

1. Turn the meter on.

2. Press * | **OK** to select **Measure.**

3. Rinse a clean tube three times with a blank. Below 1 NTU, the meter should be blanked with a 0 NTU Primary Standard or prepared turbidity free (<0.1NTU) water. For the most accurate results, use the same tube for the blanks and the sample.

4. Fill the tube to the fill line with the blank. Pour the blank down the inside of the tube to avoid creating bubbles. Cap the tube.

5. Dry the tube with a lint free cloth. Put on a dry positioning ring. Cap the tube. Wipe the tube again with a lint free cloth.

6. Open the meter lid. Insert the tube into the chamber. Align the index notch on the positioning ring with the index arrow on the meter. Close the lid.

7. Press * | **OK** to select **Scan Blank.** Remove the tube.

8. Rinse a clean tube, or the same tube three times with the standard. While the tube is inverted, wipe the lip of the tube to remove droplets trapped under the ring.

9. Fill the tube to the line with the standard. Pour the standard down the inside of the tube to avoid creating bubbles. Cap the tube.

10. Wipe the tube thoroughly again with a lint- free cloth.

11. Open the meter lid. Insert the tube into the chamber. Align the index notch on the positioning ring with the index arrow on the meter. Close the lid.

12. Press * | **OK** to select **Scan Sample.**

13. Observe the result.

14. Scroll down and press * | **OK** to select **Calibrate.**

15. Use the up or down arrows to change the highlighted digits on the display to match the concentration of the turbidity standard. Press * | **OK** to accept a digit and move to the next digit.

16. When the value on the display matches the concentration of the turbidity standard, press * | **OK** to select **Set.**

17. Press * | **OK** to proceed to Turbidity analysis.

NOTE: The meter will remember the last scanned blank reading. It is not necessary to scan a blank each time a test is performed. To use the previous blank reading, instead of scanning a new one, scroll to Scan Sample and proceed. For the greatest accuracy during the calibration procedure, be sure that after the meter is blanked, and the blank is scanned as a sample, the reading is 0.00. If not, re-blank the meter and scan the blank again until reads 0.00. When scanning the sample, scan the calibration standard three times, removing the tube from the chamber each time. The readings should be consistent.

4.4 Turbidity Analysis of Field Samples

1. Turn on the meter
2. Press * | **OK** to select **Measure.**
3. Rinse a clean tube three times with a blank. Below 1 NTU, the meter should be blanked with a 0 NTU Primary Standard or prepared turbidity free (<0.1NTU) water. For the most accurate results, use the same tube for the blanks and the sample.
4. Fill the tube to the fill line with the blank. Pour the blank down the inside of the tube to avoid creating bubbles.
5. Dry the tube with a lint free cloth. Put on a dry positioning ring. Cap the tube. Wipe the tube again with a lint free cloth.
6. Open the meter lid. Inset the tube into the chamber. Align the index notch on the positioning ring with the index arrow on the meter. Close the lid.
7. Press * | **OK** to select **Scan Blank.** Remove the tube.
8. Rinse a clean tube, or the same tube three times with the standard. While the tube is inverted, wipe the lip of the tube to remove droplets trapped under the ring.
9. Fill the tube to the line with the standard. Pour the standard down the inside of the tube to avoid creating bubbles. Cap the tube.
10. Wipe the tube thoroughly again with a lint- free cloth.
11. Open the meter lid. Insert the tube into the chamber. Align the index notch on the positioning ring with the index arrow on the meter. Close the lid.
12. Press * | **OK** to select **Scan Sample.**
13. Record the result.

4.5 Sample Dilution Procedures

If a sample is encountered that is more than 4,000 NTU, a careful dilution with 0 NTU or a very low turbidity water will bring the sample into an acceptable range. However, there is no guarantee that halving the concentration will exactly halve the NTU value. See page 21 of the operators manual for more details.

4.6 General and Annual Maintenance

Individual users are responsible for the calibration, cleaning, repair, and maintenance of the instrument.

Routine inspection and maintenance schedules vary for each piece of equipment. Typically there is minor maintenance that each piece of equipment will need to undergo prior to use in the field (such as cleaning or conditioning). Always consult the manufacturer's instructions for general and routine maintenance.

Specific per use maintenance needs for the model 2020e turbidity meter include:

1. Inspect instrument for physical damage
2. Check battery
3. Clean and dry exterior with a damp, lint free cloth.
4. Clean meter chamber and lens over LED's with a lint- free cloth slightly dampened with alcohol.
5. Scan blank
6. Turbidity verified and re-calibrated, if necessary.

DO NOT ALLOW WATER TO ENTER THE METER CHAMBER OR ANY OTHER PARTS OF THE METER.

All maintenance, repairs, and calibrations are to be documented on an equipment maintenance log or other appropriate medium. Follow the checklist provided on the equipments maintenance log for regular use maintenance needs. Any maintenance must include documentation of whether the maintenance was routine and followed the SOP or not.

Equipment logs shall be brought to the field for documenting use and calibration. The logs will be returned to the office after each field use and filed in the equipment records filing cabinet.

In the event of failure due to breakage or loss of parts, an attempt will be made to repair or replace the necessary parts by the field personnel who discover the malfunction. All repairs will be documented in field notes and/or on a non-routine maintenance log. If an instrument is rendered "out of service" or "broken", it should be tagged as such. If further repair is necessary, return the instrument to the manufacturer following proper shipping procedures.

Non-routine repairs must include documentation of the nature of the defect, how and when the defect was discovered, and any remedial action taken in response to the defect and maintained in the instrument maintenance file.

Annual maintenance is outlined in the annual maintenance checklist for each piece of equipment. All annual maintenance shall be conducted in no more than 13 month increments after the initial use of the equipment.

5.0 RESPONSIBILITIES

1. All personnel will legibly record data and observations (including phone conversations) in accordance with this SOP to enable others to reconstruct project events and provide sufficient evidence of activities conducted.

2. Prior to use and after use, all equipment will be appropriately cleaned, decontaminated and calibrated (if necessary) in accordance with the manufacturer's instructions and this SOP.

6.0 DEFINITIONS

1. *Decontamination* – Procedures followed to ensure cross contamination does not occur between sampling points or that potential contamination of equipment does not pose a hazard to sampling personnel.
2. *EPA* the U.S. Environmental Protection Agency.
3. *Maintenance* – Actions performed on equipment to standardize and/or correct the accuracy and precision of a piece of equipment to ensure that the equipment is operating within the manufacturer's specifications and standard values.
4. *Quality Assurance Unit (QAU)* any person or organizational element except the study director, designated by testing facility management to perform the duties related to quality assurance of the studies.

7.0 REFERENCES

40 CFR Part 160 Good Laboratory Practice Standards, August, 1989.

8.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

Attachment I: Manufacturer's Instruction

9.0 AUTHORIZATION

Revised by: /s/ Jim for Mike Nuss Date: 6-23-05

Michael Nuss, Staff Scientist

Approved by: _____ Date: 6-23-05

Christopher T. Stone, President

STANDARD OPERATING PROCEDURE

SEI-5.27.1

USE, MAINTENANCE AND CALIBRATION OF THE HYDROLAB MS5 WATER QUALITY MULTIPROBES

SOP Number: SEI-5.27.1
Revision Number: 1

Date Issued: 4/17/08
Date of Revision: 7/12/12

1.0 OBJECTIVE

The Hydrolab MS5 Water Quality Multiprobe is used to monitor the temperature, specific conductance, pH, oxidation-reduction potential (ORP) and dissolved oxygen concentration (DO) of water flowing through the system's flow-through cell. These parameters are measured to gain knowledge of the quality of the water and to ensure that a representative sample is being collected. To ensure the accuracy of the measurements, each of the probes in the system must be calibrated and have periodic and preventative maintenance.

2.0 POLICIES

1. The water quality parameters of the Hydrolab MS5 Water Quality Multiprobe should be verified each day before use. Maintenance, corrective actions, and calibration should be noted in the "Use, Maintenance and Calibration Log".
2. Personnel will legibly record data and observations in the field to enable others to reconstruct project events and provide sufficient evidence of activities conducted.
3. Field personnel will use a field logbook, observation and remark form (O&R) or other designated field form to record activities, measurements, and observations made in the field.
4. Any change in entries shall be made so as not to obscure the original entry. The recorder will initial corrections.
5. Field personnel will read this SOP and the appropriate sections in the Operator's Manual prior to operating the Hydrolab MS5.

3.0 SAFETY ISSUES

1. Whenever visiting a hazardous or potentially hazardous site, it is important to read, follow and sign the site health and safety plan (HASP).
2. By itself, use of the Hydrolab MS5 poses no risk to human health, however, it may be necessary to don personnel protective equipment as described in the site's HASP. In addition, protective gloves (nitrile

or latex) are recommended while handling calibration solutions as these solutions can stain skin or clothing. pH 4.01/ORP powder pillows may cause slight irritation to eyes and skin and nausea, vomiting or cramps if ingested.

3. Field visits may involve accessing remote areas. Health and safety concerns regarding these field visits are minimal however; slip, trip and fall hazards, and poisonous plant and dangerous animal, as well as getting vehicles stuck in remote areas do present considerable health and safety issues. To help ensure field staff's health and safety with remote areas, all field staff is to have daily communication with the project manager or another appropriate Stone Environmental, Inc. employee.
4. If necessary and appropriate, all chemicals are required to be received with Material Safety Data Sheets (MSDS) or appropriate application label. These labels or MSDS shall be made available to all personnel involved in the sampling and testing.

4.0 PROCEDURES

4.1 Introduction

The units owned and operated by Stone Environmental, Inc. are the Hydrolab MS5 Water Quality Multiprobes. The Hydrolab MS5 is a compact, modular system with a range of components to customize the instrument to various applications and operational modes. Components include the instrument body with a host of options, sensors, and a suite of cables for single instrument operation, network boxes and cables for network operation, external power accessories, and consumable items.

4.2 General Considerations

The Hydrolab MS5 is adversely affected by extreme temperatures and pressures. Refer to the operating manual to determine allowable operating temperatures and pressures.

4.2.1 Equipment

- Hydrolab MS5 Water Quality Multiprobe and operator's manual
- Sensors for Dissolved Oxygen, pH, ORP, and Specific Conductance
- Low Flow Cell
- Cables and power supply
- Software (Hydras 3 LT)
- Calibration solutions – one for each sensor to be calibrated, Deionized water
- Field notebook, soil boring/well log/groundwater profiling log and parameter equilibration forms, or other appropriate medium for recording field data

- Sensor Guard
- Maintenance Kit (replacement o-rings for sensors and Sonde body, spare screws with allen wrench and silicone compound)
- pH salt pellet kit (pH reference electrode/saturated KCL and AgCL)

4.2.2 Use

Water quality sensors – pH, ORP, DO, Specific Conductance – are installed in the instrument's sensor ports. Remove the multiprobe from its shipping case and inspect it for any visible damage. Connect the calibration cable (Cat. No. 013470), or detachable cable (015XXX) to the multiprobe. Align the bigger pin on the multiprobe male connector to the indicator dots on the female cable connector. Do not rotate the cable or force or twist the pins into the connectors, this can cause damage to the connector pins. Connect the power receptacle from the calibration cable and external power adapter to an approved power supply. Connect the other end of the calibration cable to the computer serial port. Start the communications program (Hydras 3 LT). The software will automatically scan for Sondes. All detected Sondes are displayed in the 'Connected Sondes' list in the main window displayed below. If a Sonde is not found, reassign the COM port (File: Options) and press **RE-SCAN FOR SONDES**. Retry until the Sonde(s) are found. Synchronize the clock on the probe with the computer clock. Be sure to condition a new DO sensor for at least half an hour, preferably overnight, before calibrating.

4.2.3 Calibration

Sensors are checked for calibration at the factory; however calibration needs to be specific for a site and application. Check the calibration prior to first use. In addition sensors should be calibrated when fouling has occurred or is noticeable, parameter measurements do not match those of a known calibrated standard and components have been added or removed for different applications or when replacing components. When Waterloo APS profiling, sondes should be calibrated before use, and at least once a week while working. If calibration is required more than once per day, a solution may be used more than once, provided it is not returned to its original container. For general calibration preparations of all sensors select a calibration standard whose values are near the field samples. Clean and prepare the sensors. To ensure accuracy of calibration discard used calibration standards appropriately. Do not reuse calibration standards (if multiple calibrations per day, solution can be reused but should be stored separate from unused solutions). It is important to maintain temperature stability during calibration, take care to keep Sonde out of direct sunlight, heat source, or other conditions that may alter the temperature of the calibration standards during calibration. To calibrate each parameter select the Calibration tab from the main window then select individual parameters

1.) pH Calibration

- Select the pH tab from the Calibration window
- Attach the calibration cup and unscrew its cap
- Thoroughly rinse the Sonde with clean water to make sure each sensor is free from contaminants.
- In a similar manner to the above step rinse the sensors twice with a small amount of the desired calibration standard
- Pour the pH standard to within a centimeter of the top of the cup.
- Enter the value of the pH standard in the field provided
- Click **CALIBRATE**. A "Calibrate Successful!" screen will be displayed.

Note: pH is a two or three-point calibration. pH 6.8 to 7.2 is treated as the "zero" all other values are "slope." First calibrate the "zero" then calibrate the "slope."

2.) ORP Calibration

- Select the ORP tab from the Calibration window
- Attach the calibration cup and unscrew its cap
- Thoroughly rinse the Sonde with clean water to make sure each sensor is free from contaminants.In a similar manner to the above step rinse the sensors twice with a small amount of the desired calibration standard
- Pour the ORP standard to within a centimeter of the top of the cup.
- Enter the value of the ORP standard in the field provided
- Click **CALIBRATE**. A "Calibrate Successful!" screen will be displayed.

3.) Specific Conductance (SpCond) Calibration

- Select one of the SpCond tabs from the Calibration window (mS/cm or μS/cm)
- Attach the calibration cup and unscrew its cap
- Thoroughly rinse the Sonde with clean water to make sure each sensor is free from contaminants.In a similar manner to the above step rinse the sensors twice with a small amount of the desired calibration standard
- Pour the SpCond standard to within a centimeter of the top of the cup.
- Enter the value of the SpCond standard in the field provided

- Click **CALIBRATE**. A "Calibrate Successful!" screen will be displayed.

Note: Specific conductance requires a two-point calibration. Calibrate the sensor to zero and then to the slope buffer. Specific Conductance is the conductivity of a substance at 25°C. When the temperature of a sample and its conductivity at that temperature are known, the software can extrapolate the conductivity to 25°C

4.) Hach LDO Sensor Calibration

- There are three methods for calibrating the Hach LDO sensor. Outlined below is the method used when water saturated air is applied to the sensor. This is the most common field method for calibrating the LDO sensor, refer to the Hach LDO Sensor Instruction Sheet for air saturated water and known calibration standards calibration methods.
- Select the LDO (SAT) tab from the Calibration window
- Attach the calibration cup and unscrew its cap
- Thoroughly rinse the Sonde with clean water to make sure each sensor is free from contaminants.Place the calibration cup with one end sealed so that the calibration cup is facing upwards. The Sonde will be inserted downwards into this cup.
- Fill the calibration cup with approximately ½ inches of deionized water or tap water (specific conductance less than 0.5 mS/cm). Water will not touch the top of the sensor cup.
- Carefully remove any water droplets from the sensor cap and temperature probe with the corner of a tissue, q-tip, or clean cotton cloth.
- Gently set the Sonde with sensors down into the calibration cup blocking any air exchange with the outside environment. Do not screw the calibration cup fully onto the Sonde as this will increase the pressure inside the calibration cup to above the barometric pressure and give false 100% saturation readings.
- Allow the dissolved oxygen and temperature readings to stabilize
- Determine the barometric pressure for entry as the calibration standard and enter into the field provided.
- Click **CALIBRATE**. A "Calibrate Successful!" screen will be displayed.
- The black coating on the LDO sensor cap wears off over time, allowing the blue LED to be visible. If more than 30% of the coating has worn off, the sensor cap should be replaced. Refer to instructions in HydroLab Manual.

4.2.4 Maintenance

Individual users are responsible for the calibration, cleaning, repair, and maintenance of the instrument.

There is minor maintenance the Hydrolab MS5 needs to undergo prior to use in the field (such as cleaning or conditioning). Always consult the manufacturer's instructions for general maintenance.

All maintenance, repairs, and calibrations are to be documented on an equipment maintenance log or other appropriate medium. Follow the checklist provided on the equipments maintenance log for regular use maintenance needs. Any maintenance must include documentation of whether the maintenance was routine and followed the SOP or not.

In the event of failure due to breakage or loss of parts, an attempt will be made to repair or replace the necessary parts by the field personnel who discover the malfunction. All repairs will be documented in field notes and/or on a non-routine maintenance log. If the instrument is rendered "out of service" or "broken", it should be tagged as such. If further repair is necessary, return the instrument to the manufacturer following proper shipping procedures.

Non-routine repairs must include documentation of the nature of the defect, how and when the defect was discovered, and any remedial action taken in response to the defect.

Detailed maintenance must be performed by a qualified technician and usually requires the shipping of the instrument to the manufacturer. Call the manufacturer for shipping instructions prior to sending the Hydrolab MS5.

General Maintenance that may be performed includes but is not limited to:

- Cleaning the Multiprobe housing
- Battery replacement
- Lithium battery replacement
- Multiprobe and sensor storage
- Electrical cable care
- Individual sensor maintenance (pH, ORP, SpCond, and Hach LDO)

Note: Consult manufactures user manual (Cat. No. 003078HY) for detailed maintenance directions.

4.2.5 Storage

For short term storage (less than 24 hours) fill the MS5 calibration cup with one inch of clean tap water and screw the cup on the multiprobe. This works for long term storage as well.

DO NOT store in pH4 solution. Prevent sensors from freezing by storing the multiprobe in conditions that freezing will not occur. Do not remove the lithium battery which powers the multiprobe internal clock. Store equipment in its designated carrying case.

5.0 RESPONSIBILITIES

1. SEI personnel must verify the Hydrolab MS5 is calibrated properly prior to use. If the values returned by the instrument are within pre-determined specifications when compared against known quantities, the instruments most recent calibration can be considered valid. Calibrations should be recorded in the Maintenance and Calibration Log.
2. It is the responsibility of SEI field staff to make sure that the Hydrolab MS5 is in proper working order prior to bringing it onto a site.
3. SEI personnel must read this SOP prior to using the Hydrolab MS5. After reading the SOP, personnel should update their personnel training folder.

6.0 DEFINITIONS

1. *EPA* the U.S. Environmental Protection Agency.
2. *Maintenance* – Actions performed on equipment to standardize and/or correct the accuracy and precision of a piece of equipment to ensure that the equipment is operating within the manufacturer's specifications and standard values.
3. *Observations & Remarks Form (O&R):* A pre-printed form, which contains mostly blank space for general note taking. The form typically prompts the user for the study or project designation, the SEI project number, the client or sponsor name, the total number of pages (page *n* of n) and requires a signature and date. The form is generally used to capture notes of one person when another, more specific form is not available.
4. *PPM*: Parts per million.
5. *Quality Assurance Unit (QAU)* any person or organizational element except the study director, designated by testing facility management to perform the duties related to quality assurance of the studies.
6. *Raw data* any laboratory worksheets, records, memoranda, notes, or exact copies thereof, that are the result of original observations and activities of a study and are necessary for the reconstruction and evaluation of the report of that study. In the event that exact transcripts of raw data have been prepared (e.g., tapes which have been transcribed verbatim, dated, and verified accurate by signature), the exact copy or exact transcript may be substituted for the original source as raw data. Raw data may include photographs, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments.

7. *Oxidation-Reduction Potential* (ORP): The ability for a compound to gain electrons (Reduce) or lose electrons (Oxidize).
8. *pH:* Negative log rhythm of Hydrogen Ion (H^+) activity in solution. Ranges from 0 (acidic) to 7 (neutral) to 14 (basic).
9. *Conductivity:* The ability of water to conduct electricity as a function of dissolve ionic species, suspended solids, and organic content. Usually measured in Siemens (S) or microsiemens (μS).

7.0 REFERENCES

1. Hydrolab DS5X, DS5 and MS5 Water Quality Multiprobes User Manual, February 2006, Edition 3

8.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

None

9.0 AUTHORIZATION

Authored by: Lee Rosberg Date: 7/13/12

Lee Rosberg, Staff Scientist

Approved by: _____ Date: 7/18/12

Seth Pitkin, Vice President

10.0 REVISION HISTORY

Revision Number 1:

1. Minor grammatical corrections.
2. Updated calibration instructions.

STANDARD OPERATING PROCEDURE

SEI-5.28.0

USE, MAINTENANCE AND CALIBRATION OF THE HACH LDO PORTABLE DISSOLVED OXYGEN METERS (HACH MODELS HQ10 AND HQ30d)

SOP Number: SEI-5.28.0
Revision Number: NA

Date Issued: 2/2/11
Date of Revision: NA

1.0 OBJECTIVE

This Standard Operating Procedure describes the use, maintenance and calibration of the Hach HQ10 and HQ30d portable luminescent dissolved oxygen (LDO) meters. The principle of operation is as follows (as per the Hach website):

The HACH LDO sensor is coated with a luminescent material. Blue light from an LED is transmitted to the sensor surface. The blue light excites the luminescent material. As the material relaxes, it emits red light. The time from when the blue light was sent and the red light is emitted is measured. The more oxygen that is present the shorter the time it takes for the red light to be emitted. This time is measured and correlated to the oxygen concentration. Between the flashes of blue light a red LED is flashed on the sensor and used as an internal reference.

2.0 POLICIES

1. The HACH LDO Portable DO Meter should be verified or calibrated as neccessary before use. Maintenance, corrective actions, and calibration should be noted in the "Maintenance and Calibration Log".
2. Personnel will legibly record data and observations in the field to enable others to reconstruct project events and provide sufficient evidence of activities conducted.
3. Field personnel will use a field logbook, observation and remark form (O&R) or other designated field form to record activities, measurements, and observations made in the field.
4. Any change in entries shall be made so as not to obscure the original entry. The recorder will initial corrections.
5. Field personnel will read this SOP prior to operating the Meter.

3.0 SAFETY ISSUES

1. If necessary and appropriate, a site-specific health and safety plan shall be created for each study site. A template for creating a proper health and safety plan is provided on the SEI network.

2. All chemicals are required to be received with Material Safety Data Sheets (MSDS) or appropriate application label. These labels or MSDS shall be made available to all personnel involved in the sampling and testing.

4.0 PROCEDURES

4.1 Equipment and Materials

1. Hach HQ10 or Hach HQ30d LDO Meter and User's Manual
2. BOD bottle or similar bottle with cap, used to create an environment of water-saturated air (LDO sensor must fit snugly into neck of bottle).
3. Replacement batteries – the LDO meter takes four AA alkaline batteries
4. Dedicated Equipment Maintenance and Calibration Log or other appropriate medium to record calibration data

Prior to use in the field the user should read and understand the manufacturer's operating instruction manual which is included in the carrying case and a copy is also provided in the equipment maintenance file.

4.2 Calibration Procedure

The LDO probe does not require calibration by the user for normal operation. However, the calibration of the LDO sensor should be checked and verified prior to use. The following steps should be followed to first verify the current calibration and, if necessary, perform recalibration in water-saturated air. (Note that these instructions are written for the HQ10 model, but when the instructions are different for the HQ30d model, those different instructions will be included in italicized font in an extra note below the applicable steps.)

1. Add ½ inch of water to the bottom of a BOD bottle or other container (Note: The container must have a cap and fit snugly around the probe. It also must allow the sensor cap to be above the water level).

2. Cap or stopper the BOD bottle and vigorously shake the bottle for 1 minute. This will saturate the entrapped air with water.

3. Press **SETUP** (same as #5 key on the HQ10) on the LDO meter.

4. Use the **UP** and **DOWN** keys to highlight "O_2 Calibration". Press **READ/OK**.

5. From the "O_2 Calibration" menu, use the **UP** and **DOWN** keys to highlight "New 100%". Press **READ/OK**.

*Note: Follow steps 3-6 if calibrating the HQ10, but if calibrating the HQ30d, replace steps 3-6 with the following: Push the **CALIBRATE** key then the **METHODS** key and then select "User Cal- 100%". Push **OK.***

6. Remove the shroud if it is installed (In Stone's case, it usually is not installed).

7. Carefully blot any liquid from the sensor surface and temperature sensor.

8. Remove the stopper from the BOD bottle and insert the probe into the 100% water-saturated air environment. Make sure that the sensor cap is above the surface of the liquid and does not get wet.

9. Press **READ/OK.**

10. While the temperature stabilizes, the meter will display "Stabilizing..." (Note: For faster stabilization, make sure the temperature of the probe is within a few degrees of the water and container temperature.) Press **POWER/CANCEL** at any time during stabilization to return to the O_2 Calibration menu.

11. After the temperature stabilizes, the percent saturation is measured and displayed using the existing calibration. If the displayed percent saturation value is acceptable (95 - 105%), press **POWER/CANCEL** to keep the existing calibration and return to the "O_2 Calibration" menu.

12. To adjust the calibration (i.e., keep the new calibration settings), press **READ/OK.** New calibration data will be displayed.

13. Press **READ/OK** to store the results of the New 100% saturation measurement.

*Note: Follow steps 9-13 if calibrating the HQ10, but if calibrating the HQ30d, replace steps 9-13 with the following: Select READ. The display will show "Stabilizing..." and a progress bar as the probe stabilizes. The display will highlight the standard value. If the value is acceptable (95 – 105%), exit the calibration menu to keep current calibration. Otherwise, select **DONE** to view the calibration summary. Select **STORE** to accept the calibration and return to the measurement mode.*

14. If necessary, the user can always revert back to the default sensor calibration for the HQ10 model, removing all user-entered adjustments made through "New 100%". To do this, follow the following steps:

    i. From "O_2 Calibration", use the **UP** and **DOWN** keys to highlight "Default". Press **READ/OK.**

    ii. Press **READ/OK** to continue. Or press the **POWER/CANCEL** key to return to the "O_2 Calibration" setup menu without changing the default calibration

iii. Again, press **READ/OK** to confirm restoration of default calibration settings and return to dissolved oxygen measurement mode. OR press the **POWER/CANCEL** key to return to the "O_2 Calibration" setup menu without changing the user-entered calibration.

iv. Verify the calibration using steps 1-11 above before use.

4.3 Analysis of Field Samples

1. Insert the LDO probe into the Stone-made custom flow through cell until it fits snugly. Teflon tape may be necessary to ensure a good leak-free seal.

2. Then use tubing to plumb the flow through cell so that it receives purge water from the profiling box or peristaltic pump outflow and so that the water then passes through the LDO flow through cell (water passing by the LDO Sensor) and into the flow cell of the water quality sonde (if a water quality sonde is also being used concurrently for measuring other physicochemical parameters).

3. Turn on the LDO meter

4. Introduce sample water flow into the flow through cell so that the LDO sensor is completely submerged and sample water can flow past the sensor cap.

5. When you are ready to take a reading, press the **READ/OK** button.

6. The meter will display "Stabilizing..." and a progress bar. As the probe stabilizes, the progress bar shrinks, then disappears when stabilization is complete.

7. Record the stabilized reading on field sampling sheet.

4.4 Handling, Storage and Maintenance

Individual users are responsible for the calibration, cleaning, repair, and maintenance of the instrument. Routine inspection and maintenance schedules vary for each piece of equipment. Typically there is minor maintenance that each piece of equipment will need to undergo prior to use in the field (such as cleaning or conditioning). Always consult the User's Manual for general and routine maintenance.

4.4.1 Handling

Take care not to touch the sensor cap with fingers, hand or any surface that may scratch or cut the cap. Also do not touch the metallic button on the side of the probe when holding it. Keep the sensor out of direct sunlight and avoid pointing directly at the sun.

If a clean water rinse alone does not remove dirt or grime from the probe, gently clean the sensor with a mild detergent, water and a soft cloth or cotton swab. Make sure not to touch the sensor or metallic button with hands or fingers and make sure that any debris present does not scratch or mar the surface of the sensor cap during cleaning. Also, do not remove the black colored substrate from the sensor cap.

If water is ever present between the sensor cap and lens, remove the cap and blot dry the sensor cap and lens with a soft dry cloth. Reinstall the sensor cap.

4.4.2 Storage

When not in use, the LDO probes should be stored ***clean and dry***. If possible, the HQ10 and HQ30d models should be stored with fresh desiccant packs in its storage case. The HQ10 LDO probe also comes with a Dry Storage Chamber. A silica gel desiccant cartridge is housed in the chamber to maintain a dry atmosphere for the sensor cap between uses. The desiccant contains a moisture indicator that changes color when it gets wet. To replace the desiccant cartridge, remove a new cartridge from its sealed bag. Open the cap on the bottom of the storage chamber and dry the inside of the chamber if moisture is present. Replace the cartridge and close the cap.

When field use is completed, the sensor should be gently rinsed with clean water and then carefully dried with a soft, lint-free cloth or kimwipe. Then insert the probe gently into the Dry Storage Chamber until it reaches the stop. If there is no dry storage chamber, the probe should be stored with fresh desiccant packs in its storage case.

The desiccant cartridge should be inspected frequently. When the cartridge color has changed, it must be either replaced or regenerated. To regenerate a saturated desiccant cartridge, first remove the saturated cartridge and replace it with a new cartridge. Place the saturated cartridge in a 105°C (221°F) drying oven overnight, or until it changes to the original color. Store regenerated cartridges in a sealed container.

4.4.3 Annual Maintenance

Sensor cap replacement is required every 365 days, or more frequently if the cap becomes damaged or fouled. The meter will keep track of the 365 days and will display a reminder message when there are 30 days of sensor service remaining on the sensor cap. Each sensor cap comes with a sensor-specific update module that will need to be replaced at the same time the sensor cap is replaced. The Hach part number for the replacement sensor cap, with update module and desiccant cartridges is 51812-00 (for the HQ10 model) and 5811200 (for the HQ30d model). Instructions for replacement will be included with the replacement sensor cap for each model.

To replace the sensor cap and update module on the HQ10 model, follow these steps:

1. Remove the old sensor cap and seal and then position the new seal on the probe body. Make sure the narrow shoulder of the seal is facing the probe tip. Slide the seal until it rests against the probe body threads at the bottom end of the threads (i.e., the end closest to the tip).
2. Finger-tighten the new sensor cap onto the probe, just until the seal is compressed. ***Do not overtighten.*** The cap will push the seal over the threads and into position. The narrow shoulder of the seal will now be inside the sensor cap.
3. Visually inspect the probe seal to make sure the narrow shoulder of the seal is inside the cap.
4. Press **SETUP.**
5. Use the **UP** and **DOWN** keys to highlight "New Sensor" in the "O_2 Setup" Menu.
6. Press **READ/OK.**
7. Now, replace the sensor-specific update module, which is provided with each replacement sensor cap. To do so, hold the update module to the back of the meter. First, slide the rounded end of the module until it stops at the probe holder on the back of the meter. The pins on the update module will align with the metal contact pads on the meter. Sensor data are loaded automatically when the meter senses the update module. The update takes less than two seconds. After the sensor data are loaded, the meter will beep twice and display the sensor lot code. Make sure the lot code on the meter display matches the lot code printed on the sensor cap.
8. Press **READ/OK** to store the new sensor setup.

5.0 RESPONSIBILITIES

1. All personnel will legibly record data and observations (including phone conversations) in accordance with this SOP to enable others to reconstruct project events and provide sufficient evidence of activities conducted.
2. Personnel will read, understand the SOP, and document that they have been properly trained prior to the use of Stone's equipment.
3. Prior to use and after use, all equipment will be appropriately cleaned, decontaminated and calibrated (if necessary) in accordance with the manufacturer's instructions and this SOP.

6.0 DEFINITIONS

1. *Decontamination* – Procedures followed to ensure cross contamination does not occur between sampling points or that potential contamination of equipment does not pose a hazard to sampling personnel.

2. *EPA* the U.S. Environmental Protection Agency.

3. *Maintenance* – Actions performed on equipment to standardize and/or correct the accuracy and precision of a piece of equipment to ensure that the equipment is operating within the manufacturer's specifications and standard values.

7.0 REFERENCES

40 CFR Part 160 Good Laboratory Practice Standards, August, 1989.

8.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

None

9.0 AUTHORIZATION

Revised by: _____ Date: 2/4/11

Morgan Greenwald, Staff Scientist

Approved by: _____ Date: 2-4-11

Christopher T. Stone, President

STANDARD OPERATING PROCEDURE

SEI-6.2.6

WATER LEVEL MEASUREMENT USE, MAINTENANCE AND CALIBRATION OF ELECTRONIC WATER LEVEL INDICATORS

SOP Number: SEI-6.2.6
Revision Number: 6

Date Issued: 04/09/93
Date of Revision: 02/20/04

1.0 OBJECTIVE

The objective of water level measurement is to determine the depth or elevation of the water table in a well. This standard operating procedure shall set forth in sufficient detail the methods, materials, and schedules to be used in the routine inspection, cleaning, maintenance, testing, and calibration, for electronic water level indicators used for water level measurement. Electronic water level meters generate and measure data, and thus must meet the requirements of 40 CFR Part 160 Subpart D.

2.0 POLICIES

1. According to 40 CFR Part 160, Subpart D, Section 160.61, Equipment used in the generation, measurement, or assessment of data and equipment used for facility environmental control shall be of appropriate design and adequate capacity to function according to the protocol and shall be suitable located for operation, inspection, cleaning, and maintenance.
2. Personnel will legibly record data and observations in the field to enable others to reconstruct project events and provide sufficient evidence of activities conducted.

3.0 SAFETY ISSUES

1. If necessary and appropriate, a site-specific health and safety plan shall be created for each study site. A template for creating a proper health and safety plan is provided on the SEI network.
2. If necessary and appropriate, all chemicals are required to be received with a Material Safety Data Sheets (MSDS) or appropriate application label. These labels or MSDS shall be made available to all personnel involved in the sampling and testing.

4.0 PROCEDURES

4.1 Decontamination

A water level indicator shall be approximately triple rinsed with distilled or de-ionized water between wells if necessary to prevent cross contamination, and at the end of use on site, unless other decontamination procedures have been outlined. If other procedures are outlined in an approved protocol or Quality Assurance Project Plan (QAPP), or special situations call for different decontamination or cleaning procedures, these procedures shall be followed.

Every effort should be made to keep the water level indicator from contact with the ground. If the water level indicator does come in contact with the ground, it shall be decontaminated prior to use.

4.2 Calibration

To calibrate a water level indicator, the indicator will be unraveled to a distance of at least 50 feet. Measurement increments will then be checked along a tape measure to ensure accuracy and that there are no missing, frayed or damaged links.

This calibration procedure shall be done after any major repair due to malfunction or breakage that may have effected the total length of the measurement cable.

Unless specified, calibration shall be done in a clean environment by a staff scientist or staff technician and documented on the appropriate water level calibration form.

4.3 Field Water Level Measurement

4.3.1 Field Equipment Needed:

1. Map of location(s) of well(s) to be measured.
2. Keys to locked wells.
3. Proper protective clothing (if necessary or as designated in the protocol or QAPP).
4. Field notebook or other acceptable recording medium.
5. Water level measuring device (electronic water indicator, interface probe, or other device).
6. Sampling gloves.
7. Decontamination materials, as necessary.
8. Clean (new) poly/plastic sheeting or other material on which well cap and other equipment may be placed.

4.3.2 Field Procedure

1. Locate wells on a well defined site map.
2. Unlock and remove the well guard if one is present, then remove the well cap. Be careful to prevent the inside of the well cap from touching the ground in order to prevent possible contamination. Clean, poly/plastic sheeting (or other appropriate material) may be spread on the ground around the well to set the cap on.
3. Determine the established measuring point, which should be marked on the well casing with indelible ink. If no mark exists, establish a measuring point at the highest point on the rim of the well casing and mark it with indelible ink. The measuring point must be on the well casing itself and not on a separate well guard, since the well guard may shift due to ground surface settling or frost heaves. If a pump head assembly is dedicated to the well, a mark should be established on the head assembly (near the access port).
4. Prior to inserting anything down the well, be sure to don clean sampling gloves and to decontaminate the measuring device.
5. Whenever possible, use an electric water level indicator to measure the water level. The water indicator consists of a graduated electric cable with a weighted probe at the end. The probe consists of an open electric connection; when the probe come in contact with the water, the connection is closed and a current travels through the wire, causing a signal to be activated. The signal may be an audible alarm or a light that is turned on. Lower the probe into the well slowly until the signal is noted. Put the graduated wire against the measurement point, then raise and lower the probe with decreasing increments to determine precisely where the water surface is.
    a. Record in the field notebook or appropriate form the water level based on the graduation of the tape; typically this is to the nearest $1/100^{th}$ of a foot
6. If a non-aqueous phase liquid (NAPL) is known or expected to be present, the measurements shall be made with an interface probe. The probe is lowered slowly into the well until a liquid is encountered. The probe signifies a low conductance liquid such as NAPL with a continuous tone. The probe is then slowly lowered deeper into the well to determine the position of the NAPL/water interface. The presence of a high conductance fluid such as water is indicated by an intermittent tone.
7. Other measuring devices, such as a measuring tape or a string, may be used to make approximate water level measurements, but they should be used only if an electronic water indicator is not available.
8. Note the water level, and the NAPL level if applicable, in a field notebook or other acceptable medium as a measurement below the top of the well casing (BTOC). Also note the time of the water level measurement.

9. Decontaminate the measuring instrument after each measurement in accordance with SOP-SEI-5.1.*n*.

10. The sequence of water level measurements shall be from least likely contaminated to most likely contaminated sampling points, or if contamination levels are unknown, from upgradient to downgradient locations, in order to reduce the potential for cross contamination.

11. Once the water level is measured, and the measurement recorded, the well cap is then set back on the well casing, and the locking well guard re-locked (if applicable). If the lock or guard needs lubrication, be sure to take appropriate steps to prevent any possible contamination of the site, well, or well casing.

4.4 General and Annual Maintenance

Individual users are responsible for the calibration, cleaning, repair, and maintenance of the water level indicator equipment.

Routine inspection and maintenance schedules vary from each piece of equipment. Typically there are minor maintenance needs each piece of equipment will need to undergo prior to use in the field (such as cleaning or conditioning). Always consult the manufacturer's instructions for general maintenance.

Specific per use maintenance needs for the water level indicator include, but is not limited to:

- Check battery/light buzzer.
- Clean probe.
- Clean tape.
- Clean outside of housing reel.
- Alconox/Liquinox rinse (refer to study requirements).
- Distilled or Deionized water rinse.
- Inspect tape for damage
- SOP followed.
- Non-routine maintenance.

The water level indicator shall be stored in a clean dry place. Care should be given to keep the instrument from dust and contamination.

All maintenance, repairs, and calibrations are to be documented on an equipment maintenance log or other appropriate medium. Follow the checklist provided on the equipments maintenance log for regular use maintenance needs. Any maintenance must include documentation of whether the maintenance was routine and followed the SOP or not.

Equipment logs shall be brought to the field for documenting use and calibration. The logs will be returned to the office after each field use and filed in the equipment records filing cabinet or within the project files.

In the event of failure due to breakage or loss of parts, an attempt will be made to repair or replace the necessary parts by the field personnel who discover the malfunction. A backup is often kept on site and may be substituted. This substitution will be documented in field notes. All repairs will be documented in field notes and/or on a non-routine maintenance log. If further repair is necessary, return the instrument to the manufacturer following proper shipping procedures. A "out of service" or "broken" tag should be placed on the piece of equipment identifying the need for maintenance and that it should not be used until further notice.

Non-routine repairs must include documentation of the nature of the defect, how and when the defect was discovered, and any remedial action taken in response to the defect.

Annual maintenance is outlined in the annual maintenance checklist for each piece of equipment. A staff technician shall perform this maintenance or other staff designated by the sampling team leader. If appropriate and necessary, all annual maintenance shall be conducted in no more than 13-month increments after the initial use of the equipment.

5.0 RESPONSIBILITIES

1. All personnel will legibly record data and observations (including phone conversations) in accordance with this SOP to enable others to reconstruct project events and provide sufficient evidence of activities conducted.

2. Prior to use and after use, all equipment will be appropriately cleaned, decontaminated and if necessary calibrated in accordance with the manufacturer's instructions.

6.0 DEFINITIONS

1. *Decontamination* – Procedures followed to ensure cross contamination does not occur between sampling points or that potential contamination of equipment does not pose a hazard to sampling personnel.
2. *EPA* the U.S. Environmental Protection Agency.
3. *FIFRA* the Federal Insecticide, Fungicide, and Rodenticide Act as amended.

4. *Maintenance* – Actions performed on equipment to standardize and/or correct the accuracy and precision of a piece of equipment to ensure that the equipment is operating within the manufacturer's specifications and standard values.

5. *Quality Assurance Unit (QAU)* any person or organizational element except the study director, designated by testing facility management to perform the duties related to quality assurance of the studies.

6. *Study* means any experiment at one or more test sites, in which a test substance is studied in a test system under laboratory conditions or in the environment to determine or help predict its effects, metabolism, product performance (pesticide efficacy studies only as required by 40 CFR 158.640) environmental and chemical fate, persistence, or residue, or other characteristics in humans, other living organisms, or media. The term "study" does not include basic exploratory studies carried out to determine whether a test substance or a test method has any potential utility.

7.0 REFERENCES

40 CFR Part 160 Good Laboratory Practice Standards, August, 1989.

8.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

None

9.0 AUTHORIZATION

Revised by: _____ Date: 2-20-04

Glenn Schellinger, Project Geoscientist

Approved by: _____ Date: 2-19-04

Christopher T. Stone, President

10.0 REVISION HISTORY

Revision number 1 (5.7.1):

1. Added Section 5.0 to detail in-house storage of water level marker.
2. Reformatted under new numbering scheme.

Revision number 2 (5.7.2):

1. Delete documentation of "use" (old section 2.1).
2. Delete sections 3.3 and 3.4 detailing annual inspections
3. Alter title to reflect that the SOP is for all of SEI's electronic water level markers and wording changed to water level "indicators".
4. Reformatted

Revision number 3 (5.7.3):

1. Removed reference to use.
2. Edited section 2.0 (General Procedure), renamed (Decontamination).
3. Removed sections 3.0 (Maintenance) and 5.0 (Storage Requirements).
4. Added section 4.0 (General and Annual Maintenance).
5. Reformatted.

Revision number 1 (6.2.1):

1. Reformatted under new numbering scheme.

Revision number 2 (6.2.2):

1. Reformatted

Revision number 3 (6.2.3):

1. Reformatted.
2. Minor wording edits.

Revision number 4 (6.2.4):

1. Added detail of measuring point for wells with dedicated sampling equipment in section 3.2.
2. Added detail on lubrication of well guard/locks in section 3.9

Revision number 5 (6.2.5):

1. Add section 3.3 on wearing sampling gloves and decontaminating measuring devices prior to lowering into a well.

SEI-5.7.3 retired, all information and procedures included in Water Level Measurement SOP SEI-6.2.6

Revision number 6 (6.2.6 Water Level Measurement, SEI-5.7.3 included in Water Level Measurements):

1. Added Use, Maintenance and Calibration of Electronic Water Level Indicators to the title.
2. Included procedures as stated in SOP SEI-5.7.3
3. Minor wording edits in Section 1.0, Objective.
4. Updated style to match SEI Style Guide – font and text. Reformatted using MS Word.
5. Added standardized section headers: 2.0 Policies, 3.0 Safety, 5.0 Responsibilities, 6.0 Definitions, 7.0 References, 8.0 Tables, Diagrams, Flowcharts and Validation data. Authorization moved to Section 9.0, andSection10.0 Revision History.
6. Deleted section on logs being given to the QAU.

STANDARD OPERATING PROCEDURE

SEI-6.3.4

SURFACE WATER SAMPLING

SOP Number: SEI-6.3.4
Revision Number: 4

Date Issued: 04/09/93
Date of Revision: 02/24/04

1.0 OBJECTIVE

The objective of this standard operating procedure (SOP) is to collect surface water samples that are representative of the surface water system. Sampling locations must be chosen in line with the objectives of the data required, and care must be taken to ensure sample integrity, and the health and safety of the sampling personnel.

2.0 POLICIES

1. According to 40 CFR Part 160, Subpart E, Section 160.81, a testing facility shall have standard operating procedures in writing setting forth study methods that management is satisfied are adequate to insure the quality and integrity of the data generated in the course of a study.
2. Personnel will legibly record data and observations in the field to enable others to reconstruct project events and provide sufficient evidence of activities conducted.

3.0 SAFETY ISSUES

1. If necessary and appropriate, a site-specific health and safety plan shall be created for each study site. A template for creating a proper health and safety plan is provided on the SEI network.
2. Care must always be taken when approaching a sampling location. Do not, under any circumstances, place yourself in danger to collect a sample.
3. If necessary and appropriate, all chemicals are required to be received with a Material Safety Data Sheets (MSDS) or appropriate application label. These labels or MSDS shall be made available to all personnel involved in the sampling and testing.

4.0 PROCEDURES

4.1 Equipment and Materials

1. Appropriate sample containers designated for the analyte of interest. Use glass or plastic containers as appropriate for the analytical procedures specified for the study. Whenever

possible, obtain certified, clean sample containers from the laboratory. If this is not possible, use clean containers and rinse them three times with water from the sample collection point before collecting the sample itself.

2. Field notebook, sample collection form, or other acceptable medium for recording field data. Ball point pens.

3. If necessary, sample collection equipment or tool for pouring water into sample containers.

4. Protective gloves if contamination is suspected in the water to be sampled, or if cold weather may be hazardous with wet hands.

5. If required, appropriate sample preservative.

4.2 Sample Collection Procedure

Prior to arrival in the field, sampling locations should be located and identified to meet the objectives of the study or project. If possible, these locations should be identified on a preliminary site map or drawing.

Put on clean protective gloves. Clean and decontaminate any sampling equipment to be used, which may include a pond dipper, glass or stainless steel beaker, weighted container, discrete sampler, or other equipment. After decontamination, rinse the equipment three times with water from the sample collection location.

If the sample container has not been pre-preserved and was not supplied by the laboratory, rinse the container three times with water from the sample collection location. This must be done before any preservative, if acquired, is added to the container.

If required, add the specified amount of preservative to the rinsed sample container and note the preservative added on the sample label.

Lower the sample collection device or the actual sample container into the water at an approximate angle of 45° so as to minimize sample aeration. If the sample is to be preserved, the sample must be collected in a container separate from the actual sample container; **do not immerse a preserved sample container in the water.**

If a separate sampling device was used to collect the sample, remove the device from the water (after triple rinsing) and decant the sample into the sample container. If the sample container contains preservative, be careful to avoid overfilling the sample container and losing the preservative.

Cap, seal, and label the sample container according to the project-specified analytical methods and the protocol.

Store the sample in a cooler with refrigerant (if appropriate) in accordance with the specified preservative for the analyte of interest.

If required by the project work plan, perform field measurements of pH, conductivity, dissolved oxygen, and/or oxidation-reduction potential using the appropriate, calibrated equipment. Wherever possible, place meter probes directly into the sample source, but where access to the source is limited, collect a representative sample in a clean, decontaminated container and take measurements immediately.

The collection of each sample will be recorded in a field logbook or on a specified sample collection form. Information to be recorded will include the following but may not be limited to:

1. Test system or Site Identification
2. Study identification or Project Identification (maybe study number)
3. Nature of the study.
4. Date of sample collection.
5. Name of personnel collecting and describing or site characterization the sample.
6. Sample identification.
7. Sample location.
8. Sample depth.
9. Sample description, including texture, color, moisture content (dry, moist, or wet), mottles (if present), and other observable features.

4.3 Sample Packing and Transportation

Package the samples in an insulated cooler or other sturdy container with refrigerant, if required. Send the samples to the laboratory for analysis as soon as is reasonable, but within the maximum holding time for the sample. If the sample is to be held in the field it must be stored at the appropriate storage temperature as required by the method or protocol. Must samples should be sent to the laboratory within three days of collection. For short holding times analytical tests such as coli form, samples must be shipped to the laboratory within six hours of collection, therefore samples may have to be hand-delivered. Chain of custody forms will be filled out and signed prior to shipment to the laboratory and included in the shipping container with the samples, according to standard operating procedures (SEI-4.2.n) and/or protocol and work plan instructions.

5.0 RESPONSIBILITIES

1. All personnel will legibly record data and observations (including phone conversations) in accordance with this SOP to enable others to reconstruct project events and provide sufficient evidence of activities conducted.

6.0 DEFINITIONS

1. *Decontamination* – Procedures followed to ensure cross contamination does not occur between sampling points or that potential contamination of equipment does not pose a hazard to sampling personnel.
2. *EPA* the U.S. Environmental Protection Agency.
3. *FIFRA* the Federal Insecticide, Fungicide, and Rodenticide Act as amended.
4. *Maintenance* – Actions performed on equipment to standardize and/or correct the accuracy and precision of a piece of equipment to ensure that the equipment is operating within the manufacturer's specifications and standard values.
5. *Quality Assurance Unit (QAU)* any person or organizational element except the study director, designated by testing facility management to perform the duties related to quality assurance of the studies.
6. *Study* means any experiment at one or more test sites, in which a test substance is studied in a test system under laboratory conditions or in the environment to determine or help predict its effects, metabolism, product performance (pesticide efficacy studies only as required by 40 CFR 158.640) environmental and chemical fate, persistence, or residue, or other characteristics in humans, other living organisms, or media. The term "study" does not include basic exploratory studies carried out to determine whether a test substance or a test method has any potential utility.

7.0 REFERENCES

40 CFR Part 160 Good Laboratory Practice Standards, August, 1989.

8.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

None

9.0 AUTHORIZATION

Revised by: _____ Date: 2/24/04

Michael Nuss, Staff Scientist

Approved by: _____ Date: 2-24-04

Christopher T. Stone, President

10.0 REVISION HISTORY

Revision number 1:

1. Reformatted under new numbering scheme.

Revision number 2:

1. Reformatted

Revision number 3:

1. Reformatted.
2. Minor wording edits.
3. Delete section 3.10 regarding description of the SOP as "general procedures" which can be altered based on analytical program.

Revision number 4:

1. Reword Objective, take out "that the sampling procedure does..." to sample integrity, and the health and safety of the sampling personnel.
2. Changed bottles to containers.
3. Updated style to match SEI Style Guide – font and text. Reformatted using MS Word.
4. Added standardized section headers: 2.0 Policies, 3.0 Safety, 5.0 Responsibilities, 6.0 Definitions, 7.0 References, 8.0 Tables, Diagrams, Flowcharts and Validation data. Authorization moved to Section 9.0, andSection10.0 Revision History.
5. Under equipment and materials, added sample preservation and if necessary, appropriate sampling device.
6. Other minor wording edits.

STANDARD OPERATING PROCEDURE

SEI-6.4.5

INSTALLATION, DEVELOPMENT AND DECOMMISSIONING OF MONITORING WELLS AND OBSERVATION WELLS

SOP Number: SEI-6.4.5
Revision Number: 5

Date Issued: 04/09/93
Date of Revision: 08/01/07

1.0 OBJECTIVE

In this Standard Operating Procedure, there is little distinction made between a monitoring well and an observation well. Unless otherwise noted, both a monitoring well and an observation well will collectively be referred to as a well.

Well construction, development and decommissioning are outlined in this procedure. A well consists of a rigid well casing with a screened interval to allow groundwater to flow into the well, and a filter pack surrounding the well screen. The well casing material, the grain size of the filter pack, and the backfill materials above the filter pack may vary considerably. In general, the main difference between a monitoring well and an observation well is that a monitoring well is sometimes constructed of inert material, such as stainless steal or Teflon-coated material, and their primary use is to access the saturated zone for groundwater sample collection. An observation well may be installed in a circumstance where groundwater levels are of interest but the collection of groundwater samples is not necessary. An observation well can also differ from a monitoring well in that it does not necessarily have a sand pack around the well screen. A clear work plan for approximate placement of the wells should be laid out and documented in the study or project files.

The purposes for installing a well may include:

1. Monitoring water levels and groundwater geochemistry
2. Mapping the local potentiometeric surfaces
3. Determining hydraulic parameters of the surficial aquifer
4. Saturated hydraulic conductivity testing.
5. Provide stratigraphic information.
6. Collecting groundwater samples.

Need for well development should be defined in the work plan on a case-by-case basis. If necessary, the procedures for well development are described below.

2.0 POLICIES

1. According to 40 CFR Part 160, Subpart E, Section 160.81, a testing facility shall have standard operating procedures in writing setting forth study methods that management is satisfied are adequate to insure the quality and integrity of the data generated in the course of a study.
2. Personnel will legibly record data and observations in the field to enable others to reconstruct project events and provide sufficient evidence of activities conducted.

3.0 SAFETY ISSUES

1. If necessary and appropriate, a site-specific health and safety plan shall be created for each study site. A template for creating a proper health and safety plan is provided on the SEI network.
2. Care must always be taken when approaching a sampling location. Do not, under any circumstances, place yourself in danger to collect a sample.
3. If necessary and appropriate, all chemicals are required to be received with a Material Safety Data Sheets (MSDS) or appropriate application label. These labels or MSDS shall be made available to all personnel involved in the sampling and testing.

4.0 PROCEDURES

4.1 Equipment and Materials

1. Hand auger, backhoe, drill rig or Geoprobe (operated by qualified personnel).
2. Well casing (screen and riser pipe).
3. Filter pack materials.
4. If required, protective well guard.
5. Field notebook, well construction log forms, or other acceptable medium for recording field notes.
6. Ruler or tape for measuring borehole depth and diameter, casing lengths, split spoon recovery, and other parameters.
7. Appropriate site personnel protective equipment as required or outlined in the Site Health and Safety Plan (e.g. hardhats, steel toed boots.)
8. If required, Photo-Ionization Detector for field screening of organic compounds.
9. Note: All drilling must be authorized by *DIG SAFE* (1-888-DIG-SAFE) and/or other appropriate agencies.

4.2 Borehole Drilling

Borings may be advanced by a variety of methods. If split spoon sampling of the soils is desired, a hollow –stem auger rig may be used. Solid core augers or other drilling techniques may be also used depending on the goals of a study. A hand auger may be used if the wells will be shallow and the soil is not prohibitively coarse.

Boreholes are most commonly drilled using hollow stem augers or by advancing bits or casing utilizing rotary boring rigs; solid stem augers may also be used. Other boring techniques may be used provided the quality and accuracy of the end product is not compromised, the variations are well documented, and the variations are approved by the on-site project manager/drilling supervisor prior to their implementation.

All augers, rods, split spoons, drilling tools or other tools that will come in contact with soil or groundwater must be cleaned at a minimum with a high temperature, high pressure steam before drilling is started. Steam cleaning should be performed after the rig has arrived on-site, but based on the potential for cross contamination, the project manager may elect to forgo on-site cleaning if it can be determined that the rig was, at a minimum, cleaned after leaving the last site the rig drilled. All actions must be documented.

On sites of known or suspected contamination, all drilling equipment that will come in contact with soil or groundwater must also be steam cleaned between boreholes to prevent cross contamination during the drilling.

The drill rig should be level, and the proper clearance between the drill rig and overhead utilities and obstructions must be maintained. Even though the drilling contractor usually establishes this, it is the responsibility of the on-site project manager/drilling supervisor to assure that the regulation and safety procedures are established.

4.3 Soil Sampling During Well Installation

Soil samples are generally collected using a split spoon sampler or other soil coring device/method. Bedrock may be sampled using a rock coring device or other appropriate means.

If required, split spoon samples are collected by driving the spoon into the soil using a hammer. The length and inside diameter of the split spoons should be recorded, and if blow counts are logged, the weight of the hammer and the dropping distance should also be recorded. The frequency of split spoon sample collection and the length of the split spoon used depend on the objectives of the investigation. The split spoon sampler should be washed with clean water prior to re-use. Detergent or steam cleaning may be required, depending on the objectives of the investigation.

Samples shall be qualitatively **described and logged** as appropriate as to:

1. Depth interval.
2. Blow counts (optional).
3. Length of sample recovered.
4. Sample description, including field texture, color, moisture content (dry, moist, or wet), mottles (if present), and other observable features.
5. Indications of contamination (if applicable).
6. Other, as appropriate.

Item 4 above may be performed at a later date if necessary. The color description should be performed in light similar to natural sunlight (if possible), and every attempt should be made to preserve field moisture conditions. If descriptions are performed at a time other than during field collection, this should be noted on the description sheet.

The collection of each sample will be **recorded** at the time of sample collection in a field log book or on a specified sample collection log form. Information to be recorded will also include the following (items italicized below may also be included to facilitate sample identification):

1. Date of sample collection.
2. Name of personnel collecting and describing the sample.
3. Sample identification.
4. Sample location.
5. Sample depth.

Samples for Laboratory Analysis: If samples are to be collected for laboratory analysis, they should be placed into a plastic sample bag, or other appropriate container, which will then be sealed and **labeled** with at least the following information:

1. Test system (sample matrix).
2. Study identification (name or study number).
3. Date of collection.
4. Sample identification number.
5. Name or initials of the sample collector.

Additional label information may be required per project specific protocol and/or quality assurance project plan.

A clear description of each sample will be noted in a field notebook, sample log form, or other acceptable medium. The description will include the details on the sample label, as well as the color, field texture, and moisture content of the sample.

Samples should be packaged securely for shipping, in accordance with standard operating procedures for sample handling, storage, and shipping.

A completed chain of custody form should accompany the samples to the laboratory, following standard operation procedures for chain of custody. A copy of the chain of custody form or list of samples transferred must be kept.

4.4 Materials

Screen and Casing. PVC is the most common well material used, but studies may require more inert materials such as stainless steel or Teflon. Polyethylene or fiberglass may be used in some situations. All joints should be made with flush-coupled threads; no glues or solvents should be used.

Annular Space. The annular space is the space between the outside of the well casing and the side of the borehole. Clean silica sand is typically used as a filter pack around the well screen. The filter pack typically extends one to two feet above the top of the screen. A bentonite seal approximately two feet thick is installed on top of the sand pack, above which the annulus may be filled either with native fill, bentonite grout (generally Portland cement with a small amount of powdered bentonite added to prevent shrinkage), bentonite slurry (a mixture of bentonite and water), bentonite chips/pellets, sand, or other material, depending on the requirements of the study. All backfill materials and depth intervals should be recorded in the field notes.

Well Guard. If required, a metal, locking well guard may be cemented into the ground surface to protect the well. A locking, PVC well cap may also be used in some cases.

State or local codes requiring different materials for installation supercede the above items. Clearly identify any changes from the above procedure.

4.5 Well Installation

When the borehole has been completed to the desired depth, the drill rods and plug are removed from the borehole and the well screen and riser pipe are lowered through the inside of the augers. The pipe should be capped or plugged at both ends to prevent the introduction of foreign objects. When the well is complete, the riser pipe should stick up above ground surface by 1-2 feet unless this would cause a hazard to vehicles or pedestrians or study needs require a different height.

Clean silica sand should be poured slowly into the annular space between the well pipe and the auger. The sand will form a filter pack around the well screen. The augers should be pulled up slowly as the sand is added to help ensure an even, continuous filter pack (If the filter pack tends to bridge, water made be poured in the annulus with the sand). The sand pack should extend from the bottom of the well up to 1-2 feet above the top of the screen.

A bentonite seal should be placed immediately above the sand pack. The seal may consist of bentonite pellets or chips, granular bentonite, bentonite slurry, or some combination thereof depending on site conditions and local code requirements. Generally, if moisture is present, granular bentonite will stick to the auger walls and cause "bridging" to occur. This problem can often be avoided by using pelletized bentonite. The plug should be at a minimum of two feet in thickness. The remainder of the annular space can be filled with bentonite grout, bentonite slurry, native material, clean silica sand, or other material, depending on the requirements of the study.

A surface plug of cement, a cement/bentonite mixture, or a bentonite/sand/native material mixture should be installed in the upper 1-2 feet of the borehole.

A locking well guard may be installed to protect the well and inhibit tampering.

State or local codes requiring different seal/backfill materials should supercede the above items. Clearly identify any changes from the above procedure.

4.6 Well Development (If Required)

After it is completed, the monitoring well should be developed to ensure that fine sediment is removed from around the well. Suspended sediment in water samples can interfere with chemical analyses. Well development also restores the natural permeability of the formation adjacent to the well and allows groundwater to flow freely into the well.

Pumping and/or surging are used to accomplish well development. Surging can be accomplished using either a surge block or reversing the pump direction to re-inject previously pumped water back down the well. If a surge block is used, ensure that the surge block is of appropriate diameter for the well and is fitted with a handle of adequate length to produce maximum water displacement and flow through the well screen. Following surging, the well would then be evacuated using either a submersible (impeller or bladder) pump, centrifugal pump, peristaltic pump or bottom discharge/filling bailer (for sediment removal). The evacuation method used will depend on well diameter, depth to water table, aquifer permeability and general yield. Depending on well conditions, it may be necessary to alternate between surging and evacuation to satisfactorily complete development.

If possible, the well will be pumped at a rate higher rate than that which would be used to purge or sample the well. By pumping the well at a higher rate, the mobilized particulates may be removed, thereby providing a cleaner well for sampling. During development, water should be removed throughout the entire water column in the well by periodically lowering and raising the pump intake.

During well evacuation, pH, specific conductivity and turbidity will be monitored and recorded every 5 minutes. Flow-through cells and/or other comparable instrumentation will be used to measure the parameters. Wells will be considered developed if the evacuated water is visibly clear and/or the parameters have stabilized to within 10 percent for three successive measurements.

The rate of flow of water should be measured and recorded during evacuation of the well. Measurements may be made using a flow meter or a watch and calibrated container such as a bucket or graduated cylinder. Evacuated water will be handled as indictated by federal or state-specific regulatory guidance depending upon the expected chemistry of the water.

The development technique used, as well as the duration of the development, volume of water removed and parameter measurement data should be recorded in a field notebook, well log form, or other acceptable medium.

4.7 Well Log

A well log should be completed with diagrams of the well construction. The following information should be included:

1. General

    a. Project designation.
    b. Well designation.
    c. Date of construction.
    d. Name of field sampling personnel.
    e. Name of drilling contractor.
    f. Drilling method (e.g. hollow stemmed auger).
    g. Inside diameter of hollow stemmed augers, if applicable.
    h. Borehole diameter or outside diameter of auger (the two should be equal).
    i. Total depth of boring.

2. Well Materials

    a. Casing diameter.
    b. Riser pipe material and length.
    c. Screen material, length, depth interval, and slot size.
    d. Sump interval, if applicable.
    e. Type of well guard.
    f. Backfill details

3. Well Development Data

    a. Date of well development.
    b. Development methods.
    c. Approximate volume evacuated from well.
    d. pH measurements.
    e. Specific Conductivity (SC) measurements.
    f. Turbidity measurements.
    g. Containment and/or disposal methods.

4. Survey and Water Level Information

    a. Top of casing elevation (optional).
    b. Ground surface elevation (optional).
    c. Length of stick-up.
    d. Water level in well (hydraulic head) at or near time of completion (if possible, this should be measured after the water level has had time to equilibrate).

All of this information should be recorded in the field at the time of well construction, except for elevation data that is not available until a survey has been completed. The information should be copied carefully from the survey onto the well log form (identify as transcribed data), or a note should be made on the form indicating that this data can be obtained from the site survey.

4.8 Water Level Measurements

A datum point at the top of the well casing should be marked with an indelible pen. This point should be the highest point on the rim of the well casing, and all water level measurements must be made from this point.

If the water levels in more than one well on a site are to be used to draw groundwater contours, the elevations of the well casings should be surveyed by an appropriate method, using the datum marks on the wells as survey points.

The location of the well should be plotted on a sketch map of the site at the time of installation. The well location may be determined by tape and compass, transit survey, or other means as appropriate.

4.9 Well Decommissioning (If Required)

Well decommissioning procedure must comply with all appropriate local and state regulations. Proper well decommissioning is necessary to:

1. Eliminate aboveground physical hazards.
2. Conserve yield and hydrostatic head of aquifers.
3. Prevent intermingling of subsurface waters.
4. Prevent contamination of the groundwater from the surface.
5. Ensure complete sealing of the borehole and prevent vertical migration of contaminants.

Depending on state and local regulations, and any prior agreements with the landowner, the manner in which a well is decommissioned and the type of equipment used for decommissioning will vary. In short, well decommissioning consists of safely extracting and removing from site, and properly disposing of all well materials, followed by permanently sealing the resulting hole.

Complete and accurate records will be kept of the decommissioning procedures to provide documentation for the study records and, if necessary, regulatory agencies. Documentation will include at least the following:

1. The project designation and/or study number.
2. The date that the well was decommissioned.
3. The personnel involved in the fieldwork.
4. Any subcontracting companies employed (drillers, backhoe operators, etc.)
5. All procedures used to remove the well materials from the borehole.
6. Any well materials left in the hole, and the approximate depths of those materials.
7. All backfill materials used, and the approximate depths of those materials.

5.0 RESPONSIBILITIES

1. All personnel will legibly record data and observations (including phone conversations) in accordance with this Standard Operating Procedure to enable others to reconstruct project events and provide sufficient evidence of activities conducted.

6.0 DEFINITIONS

1. *Study* means any experiment at one or more test sites, in which a test substance is studied in a test system under laboratory conditions or in the environment to determine or help predict its effects, metabolism, product performance (pesticide efficacy studies only as required by 40 CFR 158.640) environmental and chemical fate, persistence, or residue, or other characteristics in humans, other living organisms, or media. The term "study" does not include basic exploratory studies carried out to determine whether a test substance or a test method has any potential utility.

7.0 REFERENCES

40 CFR Part 160 Good Laboratory Practice Standards, August, 1989.

8.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

None

9.0 AUTHORIZATION

Revised by: _____ Date: 8/1/07

Gerald Edwards, PG

Approved by: _____ Date: 8/1/07

Seth E. Pitkin, Vice President

10.0 REVISION HISTORY

Revision number 1 (SEI-6.4.1):

1. Section 1.0 (Introduction): clarify purposes of monitoring wells and the designation of "observation well" in some groundwater studies.
2. Section 2.0: edit equipment list.
3. Section 3.1: make blow count recording optional; amend sample log and sample label requirements.
4. Section 3.3: make bentonite seal "approximately" two feet thick instead of "at least" two feet thick; distinguish between bentonite grout and bentonite slurry.
5. Section 3.4: change "bentonite plug" to "bentonite seal"; allow for bentonite/sand/native material mixture in surface plug.
6. Section 3.6: edit well log information.
7. Reformatted under new numbering scheme.

Revision number 2 (SEI-6.4.2):

1. Reformatted.
2. Remove inspection of rig (section 3.1).
3. Add review of state drilling codes (sections 3.3.4 & 3.4.6).
4. Add pouring water with filter pack if bridging occurs (section3.4.2).

Revision number 3 (SEI-6.4.3):

1. Adjust recommended equipment list in section 2.0.
2. Add details to section 3.1.
3. Add that state and local codes supercede construction/backfill materials per SOP.
4. Change label and logging requirements throughout section 3.2.
5. Add ability to have a stick-up less then 1 foot in section 3.4.1.
6. Delete reference to compressed air in section 3.5.2.
7. Add survey data requirements for elevations in section 3.6.2.
8. Reformatted.
9. Minor wording edits.

Revision number 1 (SEI-6.5.1):

1. Section 1.0: amend the Introduction to clarify the difference between the observation wells described in this SOP and the PVC monitoring wells that are called "observation wells" for some groundwater studies.
2. Add Section 2.0, a list of recommended equipment.
3. Add Section 3.2, split spoon sampling (cut and pasted form monitoring well SOP).
4. Section 3.3: minor edits to installation procedures.
5. Section 3.4: make the survey requirements more general.
6. Reformatted under new numbering scheme.

Revision number 2 (SEI-6.5.2):

1. Reformatted.
2. Minor wording edits.

Revision number 3 (SEI-6.5.3):

1. Reformatted.
2. Minor wording edits.
3. Adjust recommended equipment list in Section 2.0.
4. Delete Section 3.1.3.
5. Add details to Section 3.1.4 (now Section 3.1.3).

Observation Well Installation and Construction SOP included in SOP SEI-6.4.4. SOP SEI-6.5.3 retired.

Revision number 1 (SEI-6.9.1):

1. Reformatted.

Revision number 2 (SEI-6.9.2):

1. Reformatted.
2. Add discussion on sealing the borehole from the water table to the surface in Section 2.5.

Observation and Monitoring Well Decommissioning SOP included in SOP SEI-6.4.4. SOP SEI-6.9.2 retired.

Revision number 4 (SEI-6.4.4):

1. Change Title to read, "Installation, Development and Decommissioning of Monitoring Wells and Observation Wells."
2. Wording edit in Objective Section, to include references to monitoring and observation wells. Under purpose #3 add saturated. Made note about Well Development being required on a case by case basis...."
3. Minor wording edits under Equipment (i.e., hand auger). Add note on Dig Safe.

4. Change Geologic Sampling to read "Soil Sampling During Well Construction".
5. Add definition to annular space.
6. Add Section on Water Level Measurement from SOP SEI-6.5.3.
7. Add Section on Well Decommissioning from SOP SEI-6.9.2.
8. Updated style to match SEI Style Guide – font and text. Reformatted using MS Word.
9. Added standardized section headers: 2.0 Policies, 3.0 Safety, 5.0 Responsibilities, 6.0 Definitions, 7.0 References, 8.0 Tables, Diagrams, Flowcharts and Validation data. Authorization moved to Section 9.0, andSection10.0 Revision History.

Revision number 5 (SEI-6.4.5):

1. Added specificity in regard to well development in Section 4.6 Well Development.
2. Added additional well development-related data requirements to Section 4.7 Well Log, Bullet 3. Well Development Data.

STANDARD OPERATING PROCEDURE

SEI-6.16.4

HANDLING, COLLECTION AND TRANSPORTATION OF SAMPLES

SOP Number: SEI-6.16.4
Revision Number: 4

Date Issued: 11/22/93
Date of Revision: 04/16/04

1.0 OBJECTIVE

Proper handling of samples is an integral component of obtaining representative and accurate data. The handling of a sample from the time it is removed from its medium until it is placed in its designated storage area must be done with care and must be thoroughly documented. This standard operating procedure describes the basic steps to follow in proper collection and transportation of samples

2.0 POLICIES

1. According to 40 CFR Part 160, Subpart E, Section 160.81, a testing facility shall have standard operating procedures in writing setting forth study methods that management is satisfied are adequate to insure the quality and integrity of the data generated in the course of a study.
2. Personnel will legibly record data and observations in the field to enable others to reconstruct project events and provide sufficient evidence of activities conducted.

3.0 SAFETY ISSUES

1. If necessary and appropriate, a site-specific health and safety plan shall be created for each study site. A template for creating a proper health and safety plan is provided on the SEI network.
2. Care must always be taken when approaching a sampling location. Do not, under any circumstances, place yourself in danger to collect a sample.
3. If necessary and appropriate, all chemicals are required to be received with a Material Safety Data Sheets (MSDS) or appropriate application label. These labels or MSDS shall be made available to all personnel involved in the sampling and testing.

4.1 General Procedures for Handling Samples

Handling, of samples will often be outlined in an approved study protocol and/or Quality Assurance Project Plan (QAPP). If this is the case, the instructions in the protocol or QAPP will supersede this SOP.

Samples should be handled in a manner to limit any unanticipated, or unwanted exposure to temperature or pressure extremes, direct sunlight, or cross contamination, etc.

Documentation of the handling of samples should be recorded on an appropriate medium (field log book, observation and remark sheets, etc.). Documentation may include but is not limited to:

1. Sample location.
2. Sampling personnel.
3. Date of sampling.
4. Sample ID.
5. Interim storage procedure.
6. Study number.
7. Sample container description.
8. SOP or Protocol followed.

All records generated during collection and transportation of samples should be included as part of the raw data package.

A site must be adequately prepared, characterized, and instrumented according to the protocol and/or regulatory guidelines prior to application of the test substance.

A detailed site characterization shall be performed to fully identify the surface and subsurface soils, depth to groundwater, groundwater direction flow, hydraulic conductivity in the saturated and unsaturated zones, and all other parameters as outlined in an approved protocol.

All observations shall be recorded in the field notebook or on Observations and Remarks sheets.

4.2 Collection and Transportation of Soil Samples

Collection of soil samples should be documented on appropriate soil boring or sampling logs as provided for specific projects. This information should be documented as outlined in SOP SEI-6.1.*n*, SEI-6.4.*n*, SEI-6.5.*n*, and SEI-6.10.*n* or specified in an approved protocol.

If procedures for sample handling are outlined in an approved protocol, those procedures supersede any information described in this SOP. Any deviations from those procedures must be identified.

Samples should be placed into an appropriate container, and labeled according to the appropriate SOP or approved Protocol.

Samples should be kept from direct sunlight and extreme temperatures.

The samples should be protected from spillage, leakage, and cross-contamination to ensure that sample integrity is not compromised while on-site or during transport.

4.3 Collection and Transportation of Water Samples

The same procedures as described in section 3 apply to water samples. Extra precautions must be taken to ensure a good seal on each sample container to prevent the sample from leaking.

If samples are to be frozen, sample containers should be filled to allow for expansion of the sample while it freezes. Therefore, the sample container should be chosen with the desired sample size in mind.

5.0 RESPONSIBILITIES

1. All data generated during the conduct of a study, except those that are generated by automated data collection systems, shall be recorded directly, promptly, and legible in ink. All data entries shall be dated on the day of entry and signed or initialed by the person entering the data. Any change in entries shall be made so as not to obscure the original entry, shall indicate the reason for such change, and shall be dated and signed or identified at the time of the change. In automated data collection systems, the individual responsible for direct data input shall be identified at the time of data input. Any change in automated data entries shall be made so as not to obscure the original entry, shall indicate the reason for change, shall be dated, and the responsible individual shall be identified.

2. All personnel will legibly record data and observations (including phone conversations) in accordance with this Standard Operating Procedure to enable others to reconstruct project events and provide sufficient evidence of activities conducted.

6.0 DEFINITIONS

1. *Observations & Remarks Form (O&R):* A pre-printed form, which contains mostly blank space for general note taking. The form typically prompts the user for the study or project designation, the SEI project number, the client or sponsor name, the total number of pages (page *n* of n) and requires a signature and date. The form is generally used to capture notes of one person when another, more specific forms is not available.

2. *Raw data* any laboratory worksheets, records, memoranda, notes, or exact copies thereof, that are the result of original observations and activities of a study and are necessary for the reconstruction and evaluation of the report of that study. In the event that exact transcripts of raw data have been prepared (e.g., tapes

which have been transcribed verbatim, dated, and verified accurate by signature), the exact copy or exact transcript may be substituted for the original source as raw data. Raw data may include photographs, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments.

3. *Study* means any experiment at one or more test sites, in which a test substance is studied in a test system under laboratory conditions or in the environment to determine or help predict its effects, metabolism, product performance (pesticide efficacy studies only as required by 40 CFR 158.640) environmental and chemical fate, persistence, or residue, or other characteristics in humans, other living organisms, or media. The term "study" does not include basic exploratory studies carried out to determine whether a test substance or a test method has a This standard operating procedure shall be used as a guideline in ensuring that all steps have been taken in preparing the test system. ny potential utility.

7.0 REFERENCES

40 CFR Part 160 Good Laboratory Practice Standards, August, 1989.

Guidance for Prospective Ground-Water Monitoring Studies, September 16, 1998

USEPA, Pesticide Assessment Guidelines, Subdivision N, Chemistry, Environmental Fate, 1982

8.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

None

9.0 AUTHORIZATION

Revised by: _____ Date: 4-14-04

Glenn Schellinger, Project Geoscientist

Approved by: _____ Date: 4-16-04

Christopher T. Stone, President

10.0 REVISION HISTORY

Revision number 1 (SEI-6.16.1):

1. Reformatted under new numbering scheme

Revision number 2 (SEI-6.16.2):

1. Reformatted.
2. Minor wording edits.

Revision number 3 (SEI-6.16.3):

1. Reformatted.
2. Minor wording edits.
3. Delete section 3.6 detailing *requirement* of monitoring temperature of soil samples.

Revision number4 (SEI-6.16.4):

1. Titled Sections "General Procedures for Sample Handling, Collection and Transportation of Soil Samples, and Collection and Transportation of Water Samples".
2. Updated style to match SEI Style Guide – font and text. Reformatted using MS Word.
3. Added standardized section headers: 2.0 Policies, 3.0 Safety, 5.0 Responsibilities, 6.0 Definitions, 7.0 References, 8.0 Tables, Diagrams, Flowcharts and Validation data. Authorization moved to Section 9.0, andSection10.0 Revision History.

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Standard Operating Procedures
Utility Clearance for Boring Installations

Utility clearance surveys are conducted using a combination of time domain electromagnetic induction metal detection (EM61), ground penetrating radar (GPR), and precision utility location (PUL) to search for utilities and subsurface structures in the vicinity of proposed borings and monitoring wells. EM61, GPR, and PUL data are acquired in no smaller than a 20-foot by 20-foot area centered on the proposed exploration location where access allows. Where possible, multiple exploration locations are combined into larger areas. The locations of detected utilities are marked in the field with spray paint and a summary page or form is generated for each location documenting the clearance (copy attached).

Site Preparation. The areas should be clear of moveable obstructions and brush. For completeness, all available information about utilities including plans showing existing utilities, and access to utility connections must be provided. Hager-Richter can not be responsible for working in areas that are inaccessible at the time of the survey.

Grid Setup. A local grid is established centered on the location of the proposed boring. The corners of the survey grid are surveyed using GPS and are noted on the clearance summary sheets.

Data Acquisition. EM61 data and GPR data are acquired along survey lines spaced no more than 5-feet apart in the accessible portions of the survey area. The PUL equipment is used in passive mode to sweep the area of interest for utilities carrying live electrical current. If possible, connections are made to nearby utilities to determine if they enter the area of interest. The data are processed in the field and the interpretation is marked on the ground at the Site and marked on the clearance summary sheet for transmittal to the client at the end of each work day.

Equipment. *EM61*. For the EM61 survey, we will use a Geonics EM61-MK2 time domain electromagnetic induction metal detector. The EM61-MK2 is a time-domain electromagnetic induction type instrument designed specifically for detecting buried metal objects. An air-cored ½-meter by 1-meter square transmitter coil generates a pulsed primary magnetic field in the earth, thereby inducing eddy currents in nearby metal objects. The decay of the eddy current produces a secondary magnetic field that is sensed by two receiver coils, one coincident with the transmitter and one positioned 40 cm above the main coil. By measuring the secondary magnetic field after the current in the ground has dissipated but before the current in metal objects has dissipated, the instrument responds only to the secondary magnetic field produced by metal objects. Two channels of secondary response are measured in mV and are

recorded on a digital data logger. The system is generally operated by pulling the coils as a trailer with an odometer mounted on the axle to trigger the data logger automatically at approximately 8-inch intervals.

GPR. For the GPR survey, we will use our Sensors and Software Noggin Plus digital GPR system. The system includes a survey wheel to trigger recording of the data at equal intervals. We plan to use a 250 MHz antenna for this project.

PUL. We plan to use two types of Precision Utility Locating (PUL) equipment for the supplemental PUL survey. One type of the PUL instrument is a precision electromagnetic pipe and cable locator, Radiodetection RD4000 series. The RD4000 series consists of separate transmitter and receiver. The system can be used in "passive" and "active" modes to locate buried pipes by detecting electromagnetic signals carried by the pipes. In the "passive" mode, only the receiver unit is used to detect signals carried by the pipe from nearby power lines, live signals transmitted along underground power cables, or very low frequency radio signals resulting from long wave radio transmissions that flow along buried conductors. In the "active" mode of operation, the transmitter is used to induce a signal on a target pipe, and the receiver is used to trace the signal along the length of the pipe.

The second type of PUL instrument is a CST FT-70 magnetic pipe locator. This instrument is a fluxgate magnetometer that measures variations in the vertical magnetic gradient and can detect shallow subsurface metal objects.

Limitations of the Methods.

HAGER-RICHTER GEOSCIENCE, INC. MAKES NO GUARANTEE THAT ALL SUBSURFACE UTILITIES CAN BE DETECTED. HAGER-RICHTER GEOSCIENCE, INC. IS NOT RESPONSIBLE FOR DETECTING SUBSURFACE UTILITIES THAT NORMALLY CANNOT BE DETECTED BY THE METHODS EMPLOYED OR THAT CANNOT BE DETECTED BECAUSE OF SITE CONDITIONS. HAGER-RICHTER IS NOT RESPONSIBLE FOR MAINTAINING UTILITY MARK-OUTS AFTER LEAVING THE WORK AREA. FIELD MARKINGS MADE IN INCLEMENT WEATHER MAY NOT LAST.

EM61. All electromagnetic geophysical methods, including the EM method proposed here, are affected by the presence of power lines and surface metal objects (steel sided buildings, dumpsters, vehicles, railroad tracks, reinforced concrete, etc.) Where such are present, the effects of materials in the subsurface may be masked, and firm conclusions about subsurface conditions cannot be made.

Standard Operating Procedures
Utility Clearance For Boring Installations
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Detection and identification should be clearly differentiated. Detection is the recognition of the presence of a metal object, and the EM and magnetic methods are excellent for such purposes. Identification, on the other hand, is determination of the nature of the causative body (i.e., what is the body -- a drum, UST, automobile, white goods, etc.?), and EM and magnetics cannot *identify* the buried metal object.

GPR. There are limitations of the GPR technique as used to detect and/or locate targets such as those of the objectives of this survey: (1) surface conditions, (2) electrical conductivity of the ground, (3) contrast of the electrical properties of the target and the surrounding soil, and (4) spacing of the traverses. Of these restrictions, only the last is controllable by us.

The condition of the ground surface can affect the quality of the GPR data and the depth of penetration of the GPR signal. Sites covered with snow piles, high grass, bushes, landscape structures, debris, obstacles, soil mounds, etc. limit the survey access and the coupling of the GPR antenna with the ground. In many cases, the GPR signal will not penetrate below concrete pavement, especially inside buildings, and a target may not be detectable. The GPR method also commonly does not provide useful data under canopies found at some facilities.

The electrical conductivity of the ground determines the attenuation of the GPR signals, and thereby limits the maximum depth of exploration. For example, the GPR signal does not penetrate clay-rich soils, and targets buried in clay might not be detected.

A definite contrast in the electrical conductivities of the surrounding ground and the target material is required to obtain a reflection of the GPR signal. If the contrast is too small then the reflection may be too weak to recognize, possibly due to deeply corroded metal in the target, the target can be missed.

Spacing of the traverses is limited by access at many sites, but where flexibility of traverse spacing is possible, the spacing is adjusted to the size of the target.

PUL. The PUL equipment cannot detect non-metallic utilities, such as pipes constructed of unreinforced concrete, clay, ceramic, plastic, PVC when used in passive mode alone. Such pipes can be detected with the RD400, however, where surface access permits insertion of a device on which the signal can be transmitted.

The instrument also generally cannot be used to locate metal utilities located under reinforced concrete because the signal couples onto the metal rebar and mesh in the concrete, and the signal on a particular utility cannot then be traced reliably. Similarly, the method commonly cannot be used adjacent to grounded metal structures such as chain link fences and metal guardrails.

Standard Operating Procedures
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Hager-Richter Geoscience, Inc.

In congested urban areas, where several utilities are bundled or located within a short distance, the signal induced on one utility can couple onto adjacent utilities, and the accuracy of the location indicated by the instrument decreases.

Boring Clearance Summary Sheets. Boring Clearance Summary sheets will be generated for each survey area. The sheets will show at a minimum the following:

Project Name and Project Number
North Arrow (Map North)
GPS Coordinates of Grid Corners
Cultural Features
Detected Utilities and Anomalies (identified where possible)
Legend
Scale
Instrumentation Used for the Survey

The sheets will be generated for each proposed boring or survey area where multiple borings are grouped. Each area will be photo documented showing field markings after interpretation is complete.

Reporting. The locations of detected features are marked on the ground at the time of the survey with spray paint. Additionally, Hager-Richter provides one (1) paper copy and one (1) digital copy of a report of findings. The report will contain:

- Plans and/or drawings showing the locations of the geophysical survey
- EM contour plots for each of the survey areas
- Interpretation of the geophysical survey in terms of the stated objectives
- Text describing the procedures used & discussion of limitations of the methods

Typically, the written deliverable is completed approximately 3-4 weeks following the end of the field work or the receipt of all materials required from the client, whichever is later. Expedited reports and/or preliminary deliverables will incur additional costs.

STANDARD OPERATING PROCEDURE

SEI-6.19.2

USE, MAINTENANCE AND CALIBRATION OF THE IONSCIENCE PHOCHECK 1000+ PHOTOIONIZATION DETECTOR (PID)

SOP Number: SEI-6.19.2
Revision Number: 2

Date Issued: 7/19/99
Date of Revision: 02/04/2011

1.0 OBJECTIVE

A photoionization detector PID is often used to determine the presence or absence of volatile organic compounds (VOCs) in soil and water with ionization potential. Although a PID cannot necessarily differentiate between individual VOCs, it is an indispensable tool for the initial screening of a potentially hazardous site. It is therefore important to understand its operation and to keep the PID in optimal operating conditions.

The goal of soil headspace screening is to acquire preliminary information regarding volatile organic or hydrocarbon contamination in the vadose zone. The process is not meant to identify specific VOCs or hydrocarbon compounds. Rather, it will detect the relative amount of volatile organic hydrocarbons accumulated in the headspace of the sample container within the ionization potential of the lamp. This information should be used with other site-specific information to formulate an assessment of the presence or absence of contamination at the subject site.

2.0 POLICIES

1. The PID should be calibrated each day before use. Maintenance, corrective actions, and calibration should be noted in the "Maintenance and Calibration Log".
2. Personnel will legibly record data and observations in the field to enable others to reconstruct project events and provide sufficient evidence of activities conducted.
3. Field personnel will use a field logbook, observation and remark form (O&R) or other designated field form to record activities, measurements, and observations made in the field.
4. Any change in entries shall be made so as not to obscure the original entry. The recorder will initial corrections.
5. Field personnel will read this SOP prior to operating the PID.

3.0 SAFETY ISSUES

1. Whenever visiting a hazardous or potentially hazardous site, it is important to read, follow and sign the site health and safety protocol (HASP).

2. By itself, use of the PID poses no risk to human health; however, if elevated levels of VOCs are detected by the PID, it may be necessary to don personnel protective equipment as described in the site's HASP.

3. Field visits may involve accessing remote areas. Health and safety concerns regarding these field visits are minimal however; slip, trip and fall hazards, and poisonous plant and dangerous animal, as well as getting vehicles stuck in remote areas do present considerable health and safety issues. To help ensure field staff's health and safety with remote areas, all field staff is to have daily communication with the project manager or another appropriate Stone Environmental, Inc. (SEI) employee.

4.0 PROCEDURES

4.1 Introduction

The unit owned and operated by Stone Environmental, Inc. is the IonScience PhoCheck 1000+ Model PID. Although there are numerous models of PIDs available, all PIDs are based on the following principle:

> A sampling pump continuously draws air sample into the ionization chamber. The ionization chamber for the PhoCheck 1000+ PID unit is constructed as a small cavity in front of the UV lamp. The electrode is made of a wire mesh so that the high energy UV light can shine through the mesh and excite the gas molecules when they pass across the face of the UV window. The ions collected on the electrode are measured by an electrometer.
>
> A single chip microcomputer is used to control the operation of the lamp, alarm buzzer, LED, sampling pump, power supply and electrometer. It measures the electrometer readings and calculates the gas concentrations based on calibration to a known reference gas. An LCD display is used to show the readings. The user interacts with the unit though six keys on the front panel.
>
> A rechargeable lead acid battery powers the unit. A built-in charging unit allows the batteries to be charged directly from an AC power source.

4.2 General Considerations

The PhoCheck 1000+ is adversely affected by both damp and windy conditions, as well as temperature. Ensure that the PhoCheck 1000+ is used with its probe and a clean sample filter at all times. Replace the filter frequently. Refer to the operating manual to determine the lowest allowable operating temperature and humidity.

There is only one operating mode used by the PhoCheck 1000+ Model: Survey Mode. Survey mode is used for discrete VOC measurements where the peak concentration is desired. SEI will utilize the survey mode for its operations.

4.2.1 Equipment

- PhoCheck 1000+ PID and Operations Manual
- Calibration gas and regulator

- Carbon filter canister
- Spare sample filters
- Phillips head screwdriver, 7/16" wrench
- Maintenance and Calibration Log
- Clean, smooth-top jars (125 mL soil jars) and aluminum foil, or; freezer quality *zip-lock* baggies, one quart size
- Stainless steel spoon
- Distilled water
- Field notebook, soil boring/well log forms, or other appropriate medium for recording field data

4.2.2 Use

The PhoCheck 1000+ has only six controls: Power, Enter, Escape, Up Arrow, Down Arrow and Zero. The "Power" control turns the power on and off. The "Enter" control selects options and confirms settings. The "Escape" control is used to return the display to the previous screen and to abort an adjustment. Repeated pressing of this control key will return the display to the DISPLAY/FUNCTION/SELECTION screen. The "Up" and "Down" arrows allow the user to scroll up or down or to change selections. The "Zero" control zeroes out background readings.

The following provides basic instruction on the PhoCheck 1000+ menu and control functions. Once the PhoCheck 1000+ is powered on, press the "Escape" control repeatedly until the DISPLAY/FUNCTION/SELECTION screen is displayed (this screen has three icons: a graph, a number 1, and a wrench). Highlight the function menu (represented by the wrench icon) and press "Enter". Then use the "Up" and "Down" arrows to view the available functions, then press "Enter" to select the function of choice. Once a given function is selected, a cursor (highlighted symbol or number) can be moved from one selection to another by using the "Up" and "Down" keys. Pressing "Enter" will cause the selection to flash. When flashing, use the "Up" and "Down" keys to adjust its value. When you have adjusted the parameter to an acceptable level, press "Enter", which will store the adjusted parameter. In many cases, a checkmark will also appear to confirm the action. You can abort a selection by pressing the "Escape" control. Note: It is best to read the Operations Manual before attempting to operate the PID.

4.2.3 Calibration

Due to the linear output of the IonScience PID, the PhoCheck 1000+ only requires a two-point calibration. The PhoCheck 1000+ scales its linear output across a ZERO level (clean air reference) and the SPAN (a user defined gas concentration). There are two options for calibration: Factory Calibration and Custom Calibration. Factory calibration offers a safe set of calibration data. This should be used if the custom calibration fails and will keep the unit working until a good custom calibration is completed. Stone typically uses the Custom Calibration (see steps below).

The PhoCheck 1000+ is calibrated at zero using the provided carbon filter and at 100 ppm using the isobutylene calibration gas. The date and initials of the individual who performed the calibration will be documented in the maintenance and calibration log. Follow these steps for a custom calibration:

1. Select the Custom Calibration procedure from the Function Menu. A checkmark will appear to confirm the selection. Pressing "Enter" allows the user to enter the Custom Calibration menu.
2. Scroll down and select the flashing "ZERO".
3. Remove the caps from both ends of the carbon canister included in the Cal Kit Sample supplied with your PhoCheck 1000+, remove the probe and filter from the instrument and fit the carbon canister. Alternatively a known clean air supply can be used. Note – the carbon canister simply pushes onto end fitting – do not force; no turning is required.
4. Press "Enter" to start the ZERO process. A counter will count down, showing how long the carbon canister needs to remain connected.
5. At the end of the countdown, a checkmark will appear, indicating that the Zero has been accepted. Press "Escape".
6. Disconnect the carbon canister and replace the cap ends. The useful life of the carbon canister will be shortened if the canister is open to atmosphere for prolonged periods.
7. Scroll down and select the flashing "VOC".
8. The next screen shows the gas and gas concentration you have previously set up. If the information is correct, select "Enter".
9. A screen will show "VOC", a gas bottle icon and a flashing highlighted number.
10. Connect the calibration gas (Isobutylene) to the PhoCheck 1000+ and open the gas valve. Once gas is presented to the PhoCheck, press "Enter". The number will begin to count down, showing how long the instrument needs to be presented with the gas. A checkmark will appear indicating that the calibration has been accepted. "Escape" out of the calibration menu.
11. Now replace the instrument's filter and probe so PhoCheck is ready for use.
12. Carry out a Bump Test to ensure calibration was successful. In other words, expose the PID to a known concentration of gas (e.g., 100 ppm isobutylene calibration gas) and make sure it responds appropriately. Instrument is now ready for use.
13. Record value after calibrating in the Maintenance and Calibration Log.

If you are unsuccessful in calibrating the instrument, check the following points:

- The carbon canister may be contaminated
- The calibration gas may be low or empty
- If using a connector pipe, there may be a kink restricting the flow of gas

- PID lamp may require cleaning or replacement

4.2.4 Maintenance

Some tools are included with the field kit of the PhoCheck 1000+ that allow the user to perform minor maintenance. However, it is imperative that anyone attempting to clean or otherwise maintain the PID be in total comprehension of the inner controls of the PhoCheck 1000+ before opening the main body. **Please refer to the PhoCheck 1000+User Manual when performing maintenance.**

The following maintenance should be performed on a regular basis.

Maintenance	Schedule (Use dependent)
Change sample filter	Weekly
Blow out PID cell with can of computer dustoff	As needed
Replace secondary probe filter	Annually or as needed
Clean UV lamp with aluminum oxide	Annually or as needed

Detailed maintenance must be performed by a qualified technician and usually requires the shipping of the PID to the manufacturer. Stone typically sends the PID to the manufacturer annually for lamp cleaning and general maintenance – or more frequently as needed. Call the manufacturer for shipping instructions prior to sending the PID.

4.2.5 Documentation

A record of calibrations and any maintenance performed on the PID must be made in the Maintenance and Calibration log.

Typically in field use, PID values are recorded onto soil boring logs (see attached). If screening is being conducted of miscellaneous soil samples, such as those from surface samples, values should be recorded on "Observations and Remarks (O&R)" forms.

4.3 Collection of Soil Samples for Headspace Screening

Fill a clean jar or baggie approximately half full using the stainless steel spoon with a "consistent amount" of soil. Take special care to ensure that sample and container are not cross-contaminated from previous sample by rinsing spoon between samples with distilled water. It is also important to ensure that the sample is not frozen prior to analysis.

Seal top of jar with two layers of aluminum foil, or insure that bagged is properly sealed. Secure aluminum foil with either a rubber band or screw lid that fits jar. If using screw lid, do not include solid top with rubber gasket included with new mason jars.

Allow VOC concentrations to develop in the headspace of the sample container for five minutes. During this headspace development period, the sample should be shaken vigorously for a total of one minute.

Immediately pierce the aluminum foil seal (or un-zip an approximate one-inch section of baggie) with the probe of PID and note the highest (peak) reading, and sustained reading. Take care not to submerge PID tip into soil.

Record time of sample collection and peak value as ppm onto appropriate form (Soil Boring Log or O&R).

4.4 Documentation

A record of the soil screening must be made in the field at the time the peak sample headspace concentration is read from the PID. It may also be helpful to record the weather or any site-specific conditions such as excessive heat or cold that may affect the readings from the PID. Be sure that field notes are properly labeled to the project number and date.

5.0 RESPONSIBILITIES

1. Stone personnel must calibrate the PhoCheck 1000+ prior to use. Calibrations should be recorded in the Maintenance and Calibration Log.
2. It is the responsibility of Stone field staff to make sure that the PID is in proper working order prior to bringing it onto a site.
3. Stone personnel must read this SOP prior to using the PhoCheck 1000+. After reading the SOP, personnel should update their personnel training folder.

6.0 DEFINITIONS

1. *EPA* the U.S. Environmental Protection Agency.
2. *Observations & Remarks Form (O&R):* A pre-printed form, which contains mostly blank space for general note taking. The form typically prompts the user for the study or project designation, the SEI project number, the client or sponsor name, the total number of pages (page *n* of n) and requires a signature and date. The form is generally used to capture notes of one person when another, more specific forms is not available.
3. *PID*: Photoionization Detector
4. *PPM*: Parts per million.
5. *Quality Assurance Unit (QAU)* any person or organizational element except the study director, designated by testing facility management to perform the duties related to quality assurance of the studies.
6. *Raw data* any laboratory worksheets, records, memoranda, notes, or exact copies thereof, that are the result of original observations and activities of a study and are necessary for the reconstruction and evaluation of the report of that study. In the event that exact transcripts of raw data have been prepared (e.g., tapes which have been transcribed verbatim, dated, and verified accurate by signature), the exact copy or exact transcript may be substituted for the original source as raw data. Raw data may include photographs, microfilm or microfiche

copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments.

7. *VOC*: Volatile Organic Compound

7.0 REFERENCES

1. Vermont State Agency of Natural Resources, Waste Management Division "Agency Guidelines for Petroleum Contaminated Soils and Debris" August 1996.
2. Ion Science Ltd., "PhoCheck+ Insturment User Manual v2.4", November 2008.

8.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

None

9.0 AUTHORIZATION

Revised by: _____ Date: 2/4/11

Morgan J. Greenwald, Staff Scientist

Approved by: _____ Date: 2-4-11

Christopher T. Stone, President

10.0 REVISION HISTORY

Revision 1

1. Reformatted under new numbering scheme.
2. Combined with SOP draft 5.8.0. Title changed to "Use, Maintenance and Calibration of the Mini-Rae Photo Ionization Detector (PID)".
3. Added documentation and safety issues items from SOP 4.5.9.
4. Minor wording edits in Section 1.0, Objective.
5. Updated style to match SEI Style Guide – font and text. Reformatted using MS Word.
6. Added standardized section headers: 2.0 Policies, 3.0 Safety, 5.0 Responsibilities, 6.0 Definitions, 7.0 References, 8.0 Tables, Diagrams, Flowcharts and Validation data. Authorization moved to Section 9.0, and section10.0 Revision History.

Revision 2

1. Updated SOP to refer to our new PID, the IonScience PhoCheck 1000+ PID, rather than the old PID, the Mini-Rae. Changed title, any references to PID and updated all use and calibration instructions based on the PhoCheck 1000+ User Manual.

SOIL BORING LOG

Project Name: _____ Site Name: _____

SEI Study #: _____

BORING ID:	Date Drilled:	Location:
SOP followed:		
Deviations? (If YES, attach details):		
Drilling Method:		
Drilling contractor:	Inside diameter:	
Sampling method:		
Core Length: Inside diameter:		

Comments:

Depth Interval (feet bgs)	Blow Counts (per 0.5 ft)	PID (ppm)	Recovery (inches or feet)	Sample Description (color, texture, moisture, remarks)

Geoscientist:	Signature:	Date:

Geoscientist:	Signature:	Date:

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Page ____ of ____

SOIL BORING LOG (continued)			Boring ID:	SEI Study #
Depth Interval (feet bgs)	Blow Counts (per 0.5 ft)	PID (ppm)	Recovery (inches or feet)	Sample Description (color, texture, moisture, remarks)

Geoscientist:	Signature:	Date:
Geoscientist:	Signature:	Date:

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STANDARD OPERATING PROCEDURE

SEI-6.20.3

UNDISTURBED SOIL SAMPLE COLLECTION USING A THIN WALLED (SHELBY) TUBE

SOP Number: SEI-6.20.3
Revision Number: 3

Date Issued: 7/19/99
Date of Revision: 11/29/05

1.0 OBJECTIVE

When study parameters call for an undisturbed soil sample from the field suitable for soil laboratory tests (e.g. undisturbed bulk density, 1/3 water holding capacity, etc.), a thin walled sampling tube (Shelby tube) may be used. While this procedure will not necessarily produce a completely undisturbed sample (sample may smear during collection, or settle during shipment) it is an excellent method for collecting relatively undisturbed samples. The following describes the general procedure to be used when collecting thin-walled tube soil samples.

2.0 POLICIES

1. Personnel will legibly record data and observations in the field to enable others to reconstruct project events and provide sufficient evidence of activities conducted.
2. Field personnel will use a field logbook, observation and remark form (O&R) or other designated field form to record activities, measurements, and observations made in the field.

3.0 SAFETY ISSUES

1. Whenever visiting a hazardous or potentially hazardous site, it is important to read, follow and sign the site health and safety protocol (HASP).
2. Field visits may involve accessing remote areas. Health and safety concerns regarding these field visits are minimal however; slip, trip and fall hazards, and poisonous plant and dangerous animal, as well as getting vehicles stuck in remote areas do present considerable health and safety issues. To help ensure field staff's health and safety with remote areas, all field staff is to have daily communication with the project manager or another appropriate Stone Environmental, Inc. (Stone) employee.

4.0 PROCEDURES

4.1 Equipment and Materials

1. Drill rig or other appropriate machinery.

2. Thin walled tubes (Shelby tubes) - see description below.
3. Caps for tubes - may include wax.
4. Field notebook, log forms, or other recording media.
5. Labels or labeling implements.
6. Measuring tape.

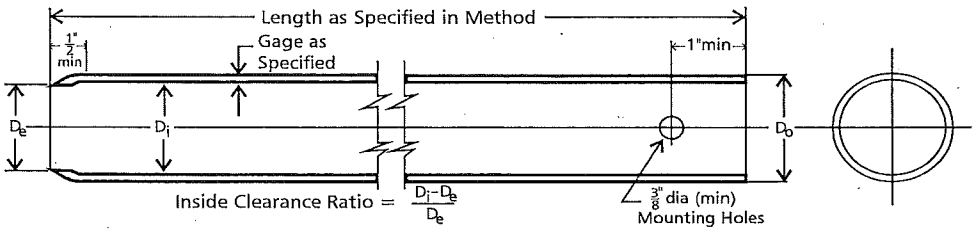

Figure 1: Example Schematic of typical dimensions of a then walled tube (ASTM D 1587-83)

4.2 Procedure

Note: Contact the drill rig operator prior to field work to discuss their typical methods and/or regional modifications to sample methods and materials. Document any differences between this SOP and practices used.

If sampling is initiated from the surface, remove plant material and other debris which may interfere with sample collection in order to ensure an undisturbed sample. If sampling at a depth below the surface where the bottom of the borehole cannot be visually inspected, clean the borehole to the sampling level by either turning the auger flights for a period of time to carry any soil up out of the path of the tube, or another method to create an undisturbed elevation for sampling. Document procedure used in the study notes.

Position the tube at the bottom of the borehole (or at the ground surface), without actually engaging the sample tube into the soil.

Advance the sampler without rotation with a steady downward motion (preferably utilizing the hydraulics of a drill rig to push the tube).

Continue the downward movement of the tube until the desired sampling depth is reached. **Do not exceed the sampling length of the usable space (tube length minus the sampler head) of the sampling tube.**

Remove the sample tube from the borehole as carefully as possible. Note if any soil falls from the tip of the tube.

Disconnect the sample tube from the driving rods and determine the length of the recovered sample. If possible, document a brief description of the soils seen at either end of the tube.

If there is any air space at either end of the sample tube, fill it with packing material such as wax or foam, then tightly cap the ends of the sample tube in a manner that will minimize movement of the soil sample within the tube.

Label the sealed sample tube with the following information (at a minimum):

1 Study/project number.

2 Date of sample collection.
3 Sample identification number (alpha-numeric label), if appropriate.
4 Depth interval.
5 Top of the interval.
6 Approximate amount of sample recovered.
7 Other, as appropriate.

Document transfer of the samples to the laboratory or courier using appropriate chain of custody forms.

5.0 RESPONSIBILITIES

1. All personnel will legibly record data and observations (including phone conversations) in accordance with this SOP to enable others to reconstruct project events and provide sufficient evidence of activities conducted.

6.0 DEFINITIONS

1. *Observations & Remarks Form (O&R):* A pre-printed form, which contains mostly blank space for general note taking. The form typically prompts the user for the study or project designation, the Stone project number, the client or sponsor name, the total number of pages (page *n* of n) and requires a signature and date. The form is generally used to capture notes of one person when another, more specific forms is not available.
2. *Raw data* any laboratory worksheets, records, memoranda, notes, or exact copies thereof, that are the result of original observations and activities of a study and are necessary for the reconstruction and evaluation of the report of that study. In the event that exact transcripts of raw data have been prepared (e.g., tapes which have been transcribed verbatim, dated, and verified accurate by signature), the exact copy or exact transcript may be substituted for the original source as raw data. Raw data may include photographs, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments.

7.0 REFERENCES

40 CFR Part 160 Good Laboratory Practice Standards, August, 1989.

8.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

None

9.0 AUTHORIZATION

Revised by: _____ Date: 11/23/05

Daniel T. Voisin, Staff Scientist

Approved by: _____ Date: 11-23-05

Christopher T. Stone, President

10.0 REVISION HISTORY

Revision 1:

1. Reformatted
2. Minor Wording edits

Revision 2:

1. Reformatted
2. Minor wording edits

Revision 3:

1. Updated style to match SEI Style Guide – font and text. Reformatted using MS Word.

   Added standardized section headers: 2.0 Policies, 3.0 Safety, 5.0 Responsibilities, 6.0 Definitions, 7.0 References, 8.0 Tables, Diagrams, Flowcharts and Validation data. Authorization moved to Section 9.0, andSection10.0 Revision History.

STANDARD OPERATING PROCEDURE

SEI-6.23.1

OBSERVATION AND MONITORING WELL SURVEYING FOR ELEVATIONS

SOP Number: SEI-6.23.1
Revision Number: 1

Date Issued: 7/19/99
Date of Revision: 11/29/05

1.0 OBJECTIVE

A field survey control program is conducted by using standard survey techniques to document piezometer and monitoring well elevations relative to a common datum or relative to each other. There are many types of leveling instruments available to conduct a survey program. This Standard Operating Procedure (SOP) describes the procedure for determining the relative elevations (altitudes) of observation and monitoring wells in the field.

2.0 POLICIES

1. Personnel will legibly record data and observations in the field to enable others to reconstruct project events and provide sufficient evidence of activities conducted.
2. Field personnel will use a field logbook, observation and remark form (O&R) or other designated field forms to record activities, measurements, and observations made in the field.

3.0 SAFETY ISSUES

1. Whenever visiting a hazardous or a study site, it is important to read, follow and sign the site health and safety protocol (HASP), if available.
2. Field visits may involve accessing remote areas. Health and safety concerns regarding these field visits are minimal however; slip, trip and fall hazards, and poisonous plant and dangerous animal, as well as getting vehicles stuck in remote areas do present considerable health and safety issues. To help ensure field staff's health and safety with remote areas, all field staff is to have daily communication with the project manager or another appropriate Stone Environmental, Inc. (Stone) employee.

4.0 PROCEDURES

4.1 Equipment and Materials

1. Level.
2. Tripod.
3. Stadia rod.

4. Field forms or other appropriate media.

4.2 Procedure

The following procedure describes a simple method to determine relative field elevations.

The method described here uses a traverse starting from a known elevation working towards unknown elevations. If there are no known elevations, then the starting point is to be given an arbitrary elevation of a temporary benchmark. The traverse will be a closed loop so that the starting point also is the ending point in the loop. This method enables determining the accuracy of the measurements by taking the difference between the initial elevation of the starting point and the final elevation of the starting point.

Surveying requires two persons, one to operate the level and one to hold the stadia rod.

1. Set up a table in the field notebook or on field data sheets with columns labeled as point location, backshot(+), foreshot(-), and elevation.
2. Set the tripod up some distance away from the starting point. (The distance depends on the ocular power of the instrument). Approximately 150 feet is a good distance.
3. Level the instrument according to the manufacturer's instructions.
4. The stadia rod is to be held vertically over the measuring point of a piezometer (monitoring well) or on a point of some known elevation. Note that this measuring point is the same point from which water levels in the piezometers (monitoring wells) are to be measured. If there is no mark, one should be made in black indelible ink. Be very careful to keep dirt or other debris from falling into the well. It may be necessary to cover the well with a plastic baggie for protection.
5. With the instrument leveled, read the measurement on the stadia at the middle cross hair. Record this measurement as the backshot (+).
6. Move the stadia rod to the next position beyond the instrument roughly the same distance as the backshot. This is designated as a turning point (could be the next piezometer or monitoring well).
7. Pivot the level around being careful not to disturb the tripod. Read the stadia rod and record as the foreshot (-).
8. Move the instrument with its tripod and level, roughly the same distance beyond the stadia rod as before (this can be done by pacing).
9. Pivot the stadia rod being careful not to change the elevation.
10. Read the measurement on the stadia rod at the turning point. Record as the backshot.
11. Repeat steps 6 through 10 working around the field to close the loop until the last foreshot is made at the original starting point.

Example of Leveling and Recording Measurements

The following example depicts the above method of leveling and recording measurements. P-1 is designated at an arbitrary elevation. P-2 is measured relative to P-1. TP is the turning point, b = backshot, f = foreshot, $I_{1,2}$ = instrument location.

1. P-1 is the starting point and is designated at an arbitrary elevation of 100.00 feet.
2. The stadia rod is at P-1. The instrument is at I_1. Read the stadia rod backshot, b = 2.00 feet. The instrument height = 100.00 + 2.00 = 102.00 feet.
3. The instrument remains at I_1. The stadia rod is moved to P-2.
4. Read the stadia rod at P-2. The foreshot is f = 0.50 feet, thus, the ground elevation at P-2 = 102.00 - 0.50 ft = 101.50 feet.
5. Move the instrument to I_2. The stadia rod remains at P-2 (which is TP1, the turning point). Read the backshot b.
6. Move the stadia rod to P-3. Read the foreshot f = 3.00.
7. Continue around the field determining the elevation of each measuring point at the piezometers and/or monitoring wells ending with the last foreshot at the starting point (P-1).
8. The calculated elevation of P-1 should be what it was assumed to be at the beginning, in this case 100.00 feet.
9. The degree of accuracy should be determined prior to the survey, and will depend on study needs. However, in most cases involving the calculation of groundwater elevations, the final foreshot should be within 0.01 feet of the starting point (P-1).

5.0 RESPONSIBILITIES

1. All personnel will legibly record data and observations (including phone conversations) in accordance with this SOP to enable others to reconstruct project events and provide sufficient evidence of activities conducted.

6.0 DEFINITIONS

1. *Observations & Remarks Form (O&R):* A pre-printed form, which contains mostly blank space for general note taking. The form typically prompts the user for the study or project designation, the Stone project number, the client or sponsor name, the total number of pages (page *n* of n) and requires a signature and date. The form is generally used to capture notes of one person when another, more specific forms is not available.
2. *Raw data* any laboratory worksheets, records, memoranda, notes, or exact copies thereof, that are the result of original observations and activities of a study and are necessary for the reconstruction and evaluation of the report of that study. In the event that exact transcripts of raw data have been prepared (e.g., tapes which have been transcribed verbatim, dated, and verified accurate by signature), the exact copy or exact transcript may be substituted for the original source as raw data. Raw data may include photographs, microfilm or microfiche

copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments.

7.0 REFERENCES

40 CFR Part 160 Good Laboratory Practice Standards, August, 1989.

8.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

None

9.0 AUTHORIZATION

Revised by: _____ Date: 11-29-05

Patrick Larsen, Project Geoscientist

Approved by: _____ Date: 11-23-05

Christopher T. Stone, President

10.0 REVISION HISTORY

Revision 1

1. Updated style to match SEI Style Guide – font and text. Reformatted using MS Word.
2. Added standardized section headers: 2.0 Policies, 3.0 Safety, 5.0 Responsibilities, 6.0 Definitions, 7.0 References, 8.0 Tables, Diagrams, Flowcharts and Validation data. Authorization moved to Section 9.0, andSection10.0 Revision History.

STANDARD OPERATING PROCEDURE

SEI-6.27.3

GROUNDWATER SAMPLING OF MONITORING WELLS

SOP Number: SEI-6.27.3
Revision Number: 3

Date Issued: 03/03/00
Date of Revision: 11/29/05

1.0 OBJECTIVE

Stone Environmental, Inc. utilizes a wide variety of sampling methods for the collection of groundwater samples from monitoring wells. This standard operating procedure outlines the sampling methods to be used for 12 volt electric submersible pumps, Grundfos pumps, bladder pumps, and bailers.

2.0 POLICIES

1. Personnel will legibly record data and observations in the field to enable others to reconstruct project events and provide sufficient evidence of activities conducted.
2. Field personnel will use a field logbook, observation and remark form (O&R) or other designated field form to record activities, measurements, and observations made in the field.

3.0 SAFETY ISSUES

1. Whenever visiting a hazardous or potentially hazardous site, it is important to read, follow and sign the site health and safety protocol (HASP).
2. Field visits may involve accessing remote areas. Health and safety concerns regarding these field visits are minimal however; slip, trip and fall hazards, and poisonous plant and dangerous animal, as well as getting vehicles stuck in remote areas do present considerable health and safety issues. To help ensure field staff's health and safety with remote areas, all field staff is to have daily communication with the project manager or another appropriate Stone Environmental, Inc. (Stone) employee.

4.0 PROCEDURES

4.1 Equipment and Materials

If using a dedicated electric submersible pump:

1. Electric submersible pump, tubing, and wiring (in place in well).
2. 12 volt power source

3. Connector cable with switch(optional)
4. Purge tubing (may be connected to calibrated buckets)
5. Replacement pump, wire, and connector kit

If using a bailer:

1. One dedicated bailer for each well is preferred. If a bailer is to be shared between wells, make sure it can be dismantled and thoroughly decontaminated in the field. Disposable bailers commonly have seams that can collect contaminants and are impossible to clean; these bailers should not be used in more than one well. Bailers must be constructed of materials that will not react with compounds to be analyzed.
2. Clean, inert string, not to be used in another well

If using a Grundfos Redi-Flo® Variable Pump assembly:

1. Grundfos Redi-Flo® Variable Pump assembly
2. Generator, ≥2500 watt with built in voltage regulator with extension cord or other power source
3. Dedicated sampling tube assembly (dedicated to each site or decontaminate between sites)
4. GPI industrial grade flow meter(see SEI SOP 5.24.*n*) with 100 ft. of tubing (optional)
5. Three, half gallon containers for decontamination procedure
6. Large tub or bucket to hold the pump and tubing/cable
7. Three containers large enough to completely immerse the Grundfos pump
8. Plastic garbage bags
9. Suspension hooks to keep tubing/cable off of the ground

If using a dedicated bladder pump:

1. Bladder pump system, including pump, tubing, and well cap
2. Oil-less air compressor compatible with the bladder pump system to be used (the compressor should be oil-less to prevent oil from collecting inside the controller and damaging it)
3. Air pressure controller for the air compressor

Equipment recommended regardless of the sampling method:

1. Protective gloves appropriate for site safety concerns and sample integrity
2. Sample containers for water quality measurements
3. Water quality meters with appropriate calibration solutions
4. Water level indicator
5. Calibrated purge buckets

6. Field notebook with protocol, applicable SOPs, study specific procedures, sampling forms, Chain of custody, and or other appropriate medium for recording field data
7. Sample containers and labels
8. Well keys
9. Calculator
10. Electrical tape
11. Clear packing tape
12. Distilled water
13. Alconox
14. Spray bottles for decontamination

4.2 Procedure

Sample Collection

Begin sample collection in locations least likely to be contaminated, and then proceed to areas more likely to be contaminated. Access the well(s).

When appropriate or necessary, all fittings, equipment, and receptacles used in the sampling process must be decontaminated in accordance with standard operating procedures for the decontamination of field equipment (see SOP 5.1.n).

Prior to measuring the water level, be sure to don appropriate, clean sampling gloves. Measure the water level in the well following the appropriate standard operating procedure for water level measurement (see SOP 6.2.n). If equipped with a dedicated pump and well head assembly, the water level indicator can be lowered into the well through the access hole in the well cap, so that the pump is not disturbed. Document use of the water level indicator on the appropriate, Use & Maintenance log following the instruments SOP.

Calculate the "well volume" - the volume of standing water in the well - using the following equation:

$$V_{gal} = (TD\text{-}WL)(\pi r^2)(CF) \qquad (1)$$

where:

V_{gal}	=	the well volume
r_{ft}	=	the radius of the well casing
π	=	3.14 (or as defined by the calculator)

TD_{ft} = the total depth of the well, measured from the top of the well casing

WL_{ft} = the water level in the well, measured from the top of the well casing

CF = conversion factor (cubic ft. to US gal. = 7.48gal/ft^3)

The $[(\pi r^2)(CF)]$expression in the well volume formula may already be calculated. For example, the attached sampling form has been calculated for a 2 inch diameter well.

Note that if the well is equipped with a dedicated pump, this equation does not take into account the volume displaced by the pump, so it will over-estimate the actual volume of water in the well. Document all well volume calculations on the sampling form or in another appropriate medium. Refer to SOP 4.5.*n* for more information on documentation of raw data. If appropriate, calculate 1, 3, and 5 well volumes for each well to be purged.

The standing water in the well has been out of direct contact with the soil and exposed to the open air. The effects of this on the quality of the water are difficult to determine, but to avoid any question of whether the sample is representative of true groundwater quality on the site, it is best to thoroughly purge the well before collecting a groundwater sample. Determine which type of sampling apparatus is to be used and follow the corresponding purging procedure from the following list.

Dedicated electric submersible pump:

1. Connect the purge tubing to the existing outlet extending above the well cap. Run the tubing from the outlet to the calibrated bucket.
2. Open the valve on the well sampling assembly (if one exists).
3. Connect the power supply to the pump leads using the switch box and connector cable and turn the switch on. If you do not have the switch box, connect the leads to complete the circuit when you are ready to begin pumping.
4. Confirm that water is flowing into the bucket via the purge tubing, if no water is flowing then turn the pump off and check connections.
5. Continue purging until complete as defined below.

Bailer:

1. Put on clean sampling gloves
2. Obtain a clean bailer. Do not allow the bailer to come into contact with surface soils on the site
3. Attach the inert string to the bailer. Make sure the knot is secure and that there is a sufficient length of string to reach the bottom of the well.
4. Lower the bailer into the well on the nylon string, and allow the bailer to fill with water.
5. Raise the bailer out of the well, coiling the string so as not to let it come into contact with the ground
6. Dump the bailer into one of the calibrated buckets.

7. Repeat steps 2-6 until purging is complete as defined below.

Grundfos Redi-Flo® Variable Pump:

1. Start the generator, allow it to warm up for at least 5 minutes, and check generator settings, 115 volts AC±10%, optimum setting of 120 volts without load.
2. Rinse pump with alconox and distilled water
3. Connect the sample tubing to the pump taking care not to let it come in contact with the ground. If the tubing is on a reel, it may be helpful to put a clean plastic bag under the reel to avoid contact with the ground.
4. Carefully lower the pump into the well taking care that is does not rest on the bottom.
5. Plug the Redi-Flo Variable Frequency Device (VFD) into the generator.
6. Using the mode selection dial, choose the pump model.
7. Turn the ten-turn speed dial fully counter clockwise.
8. Connect the pump motor lead to the VFD.
9. The frequency display should read 0.0, if it does not refer to the Redi-Flo Installation and Operating instructions.
10. If the unit has not been used for more than six months, allow a five minute warm up period before starting the pump.
11. If using the GPI industrial grade flow meter, connect the flow meter to the dedicated sample tubing and run the purge tubing down gradient of the well. If a flow meter is not used, calibrated buckets or some other purging method can be used.
12. Start the pump by switching the Redi-Flo VFD run/stop/reset switch to run
13. Adjust the pump to the desired speed using caution to preclude pumping the well dry. It may be useful to record the optimum pumping rate in hertz in the comments section of the applicable Monitoring Well Sampling form.
14. Purge the well until complete as defined below.

Dedicated bladder pump:

1. Connect the purge tubing to the existing outlet extending above the well cap.
2. Run the tubing from the outlet to the purge bucket.
3. Using the two air hoses, connect the compressor to the air pressure controller and the air pressure controller to the air tube inlet on the well cap. Set the controller to regulate the air pressure used for pumping and turn on the controller (if necessary/ applicable).
4. Start the air compressor, and if necessary/applicable, open the valve to allow the air to flow to the controller.

5. Confirm that water is flowing to the purge buckets via the purge tubing, if not, check all connections and turn the pump off if checking connections does not solve the problem.

6. Continue until purging is complete as defined below.

Track the volume purged. If you are using calibrated buckets, empty the buckets off-site down gradient of all wells in the study. Take care not to let the purge water flow or spill onto the ground around the well.

Remove a volume of water equal to or greater than 3-5 times the well volume. Water quality parameters may be monitored to assist in determining if the well has been adequately purged. The pH, temperature, and conductivity of the groundwater (and / or other parameters, depending on the requirements of the study), can be monitored while the well is purged using the following procedure:

1. After purging at least one well volume. Collect a sample of the purge water in a beaker or a clean container (other than the sample bottle) and measure and record the water quality parameters.

2. Continue to purge and collect water quality parameters at intervals of ≥ **0.5** well volumes.

3. If after **three** consecutive water quality parameter measurements **and** after purging a minimum of **three** well volumes, the parameters for temperature and conductivity vary less than 10% and the pH varies less than 0.1 unit, purging can be halted.

4. If any one of the parameters are outside of the range, successive measurements of water quality can than be made until the parameters stabilize per the above ranges. If the parameters do not stabilize after **five** well volumes have been purged, purging may be halted.

If water quality meters are used be sure to document their use on the appropriate, Use & Maintenance log following the instruments SOP. If calibrations are required, refer to the instruments SOP or the manufacturers instructions. Record the method of purging, the parameters monitored, if applicable, and the approximate volume of water purged. If any of the water quality meters are malfunctioning or are not used, always attempt to purge five well volumes. Regardless of whether water quality parameters are measured, a **minimum of 3 well volumes must always be purged.** The exception to this is if the well is purged dry.

Avoid purging the well completely dry (especially if sampling for volatiles). If the well is purged dry, allow the well to re-charge adequately enough to permit collection of a sample. **Note on the monitoring well sampling form if the well was purged dry** (purge volume may be less than 3 well volumes in this case).

Put on clean sampling gloves. Collect the groundwater sample in a clean bottle or other container appropriate for the analytical method to be used. The sample is collected by carefully removing the cap from the sample bottle, taking care not to touch the inside of the cap or bottle with your fingers, the discharge tube, or any other equipment. Do not put the cap down. Fill the sample bottle according to the requirements for the analysis (rinse as required by the protocol). Tightly cap the bottle. The sample container should be labeled, at a minimum, with the following information. (Generally, it is easier to attach the label or write on the container before collecting the sample. Condensation on the containers forms after sample collection, and reduces the likelihood of tape or ink sticking):

1. Study number(s) or project name

2. Sample identification (including the type of sample - groundwater)

3. Date of sample collection

Secure the label with a wrap of clear tape if applicable. Follow protocol or Quality Assurance Project Plan requirements for further label needs.

If appropriate to the study, the cap of the bottle can be secured with tape to prevent accidental loss of sample during storage and shipment.

Place the samples out of direct sunlight as soon as possible or into the appropriate storage devices such as a cooler or boxed container.

4.3 Documentation

A record of the sample collection must be made in the field at the time the sample is collected. The record should be made in a field notebook, a sample collection form, or other medium acceptable according to the study protocol or work plan. The information to be recorded must include the following:

1. Date and time of sample collection.
2. Name(s) of the personnel performing the sampling.
3. Sample location, well identification, and / or sample identification.
4. Project/study designation.
5. Water level prior to purging (equilibrium level).
6. Well volume calculations.
7. Volume purged from the well prior to sampling.
8. Method of sample collection.
9. Chemical parameters monitored in the field (if applicable).

4.4 Packing and Transportation

Package the samples in an insulated cooler or other sturdy container with ice or ice substitute if required. Send the samples to the laboratory for analysis as soon as is reasonable, but well within the maximum holding time for the sample allowing for time in transit. A completed Chain-of-Custody Form must accompany the sample set, in accordance with standard operating procedures for chain of custody forms.

4.5 Equipment Maintenance, Calibration and Decontamination

Follow Stone SOPs for specific equipment to determine the appropriate equipment, maintenance, calibration and decontamination procedures,

Stone does not maintain 12V electric submersible pumps. Should one fail, it will be replaced with a new one. Replacement of a pump should be noted in files and the number of the pump should be recorded.

4.6 Procedure for Decontamination of the Grundfos Pump and Reel

After use of this equipment this decontamination procedure must be followed prior to sampling another well. Ideally, decontamination will occur at the site before storing the equipment for any period of time.

1. Once the well has been purged and/or sampled, place the tubing/cable and pump into a large bucket lined with a clean garbage bag.
2. Leaving the reel attached to the well "walk" the bucket away from the well until the total length of tubing/cable that had been inside the casing is laid out for washing. In order to keep the tubing/cable off the ground after it has been cleaned it may be necessary to suspend the tubing/cable, with a series of hooks for example.
3. Spray the entire length of the tubing/cable with alconox being careful to coat each side thoroughly.
4. Spray the entire length again with distilled water until the alconox has been completely rinsed off.
5. Put on clean gloves.
6. Reel the tubing/cable up taking care not to let it come in contact with anything.
7. Place the pump into a container filled with alconox solution. Run the pump such that approximately two gallons of alconox "clear" the pump and associated tubing.
8. Place the pump into another container filled with distilled water and again run approximately two gallons through the system.
9. Place the pump in a third container and repeat step 9.
10. Unscrew the plug in the bottom of the pump and rinse well with distilled water. Fill the small space with distilled water and replace screw.
11. Replace the plug on the reel casing so that nothing can enter the tubing. Some distilled water may remain in the tubing that will be evacuated next time the pump is started.
12. Document the equipment has been decontaminated on the Use & Maintenance log.

5.0 RESPONSIBILITIES

1. All personnel will legibly record data and observations (including phone conversations) to enable others to reconstruct project events and provide sufficient evidence of activities conducted.

6.0 DEFINITIONS

1. *Observations & Remarks Form (O&R):* A pre-printed form, which contains mostly blank space for general note taking. The form typically prompts the user for the study or project designation, the Stone project number, the client or sponsor name, the total number of pages (page *n* of n) and requires a signature and date. The form is generally used to capture notes of one person when another, more specific forms is not available.

2. *Raw data* any laboratory worksheets, records, memoranda, notes, or exact copies thereof, that are the result of original observations and activities of a study and are necessary for the reconstruction and evaluation of the report of that study. In the event that exact transcripts of raw data have been prepared (e.g., tapes which have been transcribed verbatim, dated, and verified accurate by signature), the exact copy or exact transcript may be substituted for the original source as raw data. Raw data may include photographs, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments.

7.0 REFERENCES

40 CFR Part 160 Good Laboratory Practice Standards, August, 1989.

8.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

Attachment 1: Example Monitoring Well Sampling Form

9.0 AUTHORIZATION

Revised by: _____ Date: 10-29-05

Patrick Larsen, Project Geoscientist

Approved by: _____ Date: 11-23-05

Christopher T. Stone, President

10.0 REVISION HISTORY

Revision 1 and 2:

1. Reformatted
2. Added some items for decontamination to Section 2.0, #3
3. Minor word changes to Section 3.1.5
4. Deleted requirement for rinsing the sample bottles prior to collection.
5. Added Section 3.5 Procedures for Decontamination of the Grundfos Pump and Reel

Revision 3:

1. Updated style to match Stone Style Guide – font and text. Reformatted using MS Word.
2. Added standardized section headers: 2.0 Policies, 3.0 Safety, 5.0 Responsibilities, 6.0 Definitions, 7.0 References, 8.0 Tables, Diagrams, Flowcharts and Validation data. Authorization moved to Section 9.0, andSection10.0 Revision History.

Attachment I
Example Monitoring Well Sampling Form

MONITORING WELL SAMPLING FORM STONE ENVIRONMENTAL INC

Study Title	
Sponsor	
Protocol Number	
Study Director	
Stone Environmental, Inc. Study Number	

SITE ID	
Date	
SSP Followed	
Sampling Method	Whale Pump Bailer (Circle the appropriate one)
Sampling Personnel	
Weather	

Calculate Purge Volumes

Time of level measurement:	

Total Well Depth (btoc) — Depth to Water (btoc) = Height of Water Column

Well Water Volume

_____ feet – _____ feet = _____ feet X 0.169 gal/feet = _____ gallons

3 X Well Water Volume	gallons
5 X Well Water Volume	gallons

Time (HR:MIN)	pH (su)	Conductivity (mmho/cm)	Temperature (°C)	Cumulative Volume (gallons)

Total Volume Purged: _____ Gallons

Sample Number	Time Collected (HR:MIN)	Approx. Vol. (mL)	Comments

Signature of Sampler:		Date:	

Note: BTOC = Below Top of Casing

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STANDARD OPERATING PROCEDURE

SEI-6.34.0

PROCEDURE FOR SAMPLING GROUNDWATER MONITORING WELLS USING LOW STRESS (LOW FLOW) TECHNIQUE

SOP Number: SEI-6.34.0
Revision Number: NA

Date Issued: 01/21/05
Date of Revision: NA

1.0 OBJECTIVE

This Standard Operating Procedure (SOP) provides a framework for collecting groundwater samples that are indicative of mobile organic and inorganic loads at ambient flow conditions (both the dissolved fraction and the fraction associated with mobile particulates). The SOP emphasizes the need to minimize stress by low water-level draw downs, and low pumping rates (usually less than 1 liter/min) in order to collect samples with minimal alterations to water chemistry. This SOP is aimed primarily at sampling monitoring wells that can accept a submersible pump and have a screen, or open interval length of 10 feet or less. Samples thus obtained are suitable for analyses of ground water contaminants (volatile and semi-volatile organic analytes, pesticides, PCBs, metals and other inorganics), or other naturally occurring analytes..

2.0 POLICIES

It is the policy of Stone Environmental Inc. (Stone) that all field staff conducting this sampling read this SOP prior to acquiring samples. Refer to SEI 6.16.4 for sample handling, preservation and transportation procedures.

Stone personnel will legibly record data and observations in the field to enable others to reconstruct project events and provide sufficient evidence of activities conducted. Field personnel will use a field logbook, observation and remark form or other designated field form to record activities, measurements, and observations made in the field.

3.0 SAFETY ISSUES

The corporate Health and Safety Plan and the Site Health and Safety Plan specify the procedures to be followed and equipment to be used during site activities. The following is a brief and general overview of safety issues.

- Potential Safety Issues include:
    - Heavy equipment (drill rigs or direct push rigs) hazards
    - Overhead utility hazards

- Underground utility hazards
- Traffic/ motor vehicle hazards
- Hazards associated with exposure to various chemicals
- Slip, trip and fall hazards
- Pinch point hazards
- Compressed gas hazards
- Fire hazards from hot work (e.g., grinding)
- Gasoline hazards (filling generator)

- SEI staff and others under contract with SEI that may be present on-site will wear a hardhat whenever overhead hazards are present.
- Appropriate eye protection should be worn
- Hearing protection shall be worn whenever the drill rig is actively advancing profiling equipment.
- Steel toed boots will be worn
- SEI staff will read the site health and safety plan (HASP) prior to beginning a project.

Additional personal protective equipment shall be worn in accordance to the health and safety document.

4.0 PROCEDURES

Stabilization of indicator field parameters is used to indicate that conditions are suitable for sampling to begin. Achievement of turbidity levels of less than 5 NTU and stable draw-downs of less than 0.3 feet, while desirable, are not mandatory. Sample collection may still take place provided the remaining criteria in this procedure are met. If after four (4) hours of purging, indicator field parameters have not stabilized, one of the following three courses of action may be taken: a) discontinue purging, collect samples and provide full explanation of attempts to achieve stabilization (note: there is a risk that the analytical data obtained, especially metals and strongly hydrophobic organic analyses, may not meet the sampling objectives), b) continue purging until stabilization is achieved, c) discontinue purging, do not collect any samples, and record in study or project records that stabilization could not be achieved (documentation must describe attempts to achieve stabilization).

4.1 Equipment

1. Extraction device. Adjustable rate, submersible pumps should be used (for example, centrifugal or bladder pump constructed of stainless steel or Teflon
2. Tubing. Fluorinated Ethyl Propylene (FEP) will be used when sampling is to include VOCs, SVOCs, pesticides, PCBs and inorganics. Stainless steel tubing may be used when sampling for VOCs, SVOCs, pesticides, and PCBs. However, it should be used with caution when sampling

for metals. The use of 1/4 inch or 3/8 inch (inner diameter) tubing is preferred. This will help ensure the tubing remains liquid filled when operating at very low pumping

3. Slope Indicator or other water level measuring device. Water level measuring device(s), capable of measuring to 0.01 foot accuracy (electronic "tape", pressure transducer). Recording pressure transducers, mounted above the pump, are especially helpful in tracking water levels during pumping operations, but their use must include check measurements with a water level "tape" at the start and end of each record
4. Graduated cylinder or other volumetric lab ware to determine purge volume.
5. Power source (generator, nitrogen tank, etc.) appropriate for pump. If a gasoline generator is used, it must be located downwind and at least 30 feet from the well so that the exhaust fumes do not contaminate the samples
6. Indicator field parameter monitoring instruments (pH, oxidation-reduction potential (ORP), dissolved oxygen (DO), turbidity, specific conductance, and temperature). Use of a flow-through cell is required when measuring all listed parameters. Standards to calibrate the water quality instrument should be brought to the site for calibration prior to use. Refer to the manufacturer's operation manual for maintenance and calibration procedures for the water quality instrument and to SEI 10.7.1 operation of the YSI 600 XL.

4.2 Preliminary Site Activities

1. Check for security damage or evidence of tampering on each well, record pertinent observations
2. Remove well cap and immediately measure VOCs at the rim of the well with a PID or FID instrument and record the reading in the logbook
3. If the well casing does not have a reference point (usually a V-cut or indelible mark in the well casing), make one. Describe its location and record the date of the mark in the logbook.
4. Check newly constructed wells for the presence of NAPL before the initial sampling round. Note: Procedures for collection of LNAPL or DNAPL samples are not addressed in this SOP.

4.3 Purging and sampling procedures

Sampling wells in order of increasing chemical concentrations (known or anticipated) is preferred. If applicable, refer to the site Quality Assurance Project Plant (QAPP) for additional sample information including frequency, preservation methods, containers, and specific analytical methods.

4.3.1 Install Pump

Lower pump and any safety cables, tubing and/or electrical lines slowly (to minimize disturbance) into the well to the midpoint of the zone to be sampled. In most instances, the pump will be positioned in the middle of the well screen. For cases where the well screen straddles the water table, the pump should be position 2 feet above the bottom of the well.

4.3.2 Measure Water Level

Before starting pump, measure water level. If a recording pressure transducer is used, initialize starting condition. After recording the height of the water column in the well, determine the volume in gallons by the following formula:

Volume in gallons = (Height of water column * π*Radius^2)*7.4805

When using the above formula, all measurements (height and radius) must be in feet.

4.3.3 Purge Well

4.3.3.1 Initial Low Stress Sampling Event

Start pump at its lowest speed setting and slowly increase the speed until discharge occurs. Check water level. Adjust pump speed until there is little or no water draw-down (less than 0.3 feet). If the minimal draw-down that can be achieved is greater than 0.3 feet, continue purging until indicator field parameters stabilize

Monitor and record water level and pumping rate every three to five minutes (or as appropriate) during purging. Record any pumping rate adjustments (both time and flow rate). Pumping rates should, as needed, be reduced to the minimum capabilities of the pump to ensure stabilization of indicator parameters. Adjustments are best made in the first fifteen minutes of pumping in order to help minimize purging time. During pump start-up, drawdown may exceed the 0.3 feet target and then "recover' as pump flow adjustments are made.

Using the appropriate volumetric container (one that is scalable relative to the volume to be purged), measure the volume of water purged at the same interval as field parameters are recorded (Section 4.3.4). If water is or believed to be contaminated, purge water must be containerized as specified in the site work plan. Purge volume calculations should utilize stabilized drawdown value, not the initial drawdown. Do not allow the water level to fall to the intake level (if static water level is above the well screen, avoid lowering the water level into the screen). The final purge volume should be greater than the stabilized drawdown volume plus the extraction tubing volume. This may not be possible in wells with low recharge rates.

Wells with low recharge rates may require the use of special pumps capable of attaining very low pumping rates (bladder, peristaltic) and/or the use of special techniques. If the recharge rate of the well is lower than the pump's minimum extraction rate, then continue to purge until the drawdown is near (but not below) the top of the well screen. Once the drawdown is within six (6) inches of the top of the well screen, discontinue purging. The well should be sampled as soon as the water level has recovered sufficiently to collect the appropriate volume needed for all anticipated samples (the intake should not be moved during this recovery period). In this case only, samples may be collected even if the well was not purged for four (4)

hours and the indicated field parameters did not stabilize (as instructed in Section 4.0).

4.3.3.2 Subsequent Low Stress Sampling Events

Prior to sampling, check intake depth and drawdown information from previous sampling event(s) for each well. Duplicate, to the extent practicable, the intake depth and extraction rate (use the final pump dial setting information) from previous event(s). Perform purging operations as above

4.3.4 Monitor Indicator Field Parameter

During well purging, monitor indicator field parameters (turbidity, specific conductance, pH, ORP, DO) every 3 to 5 minutes. Values are to be recorded on appropriate field forms or on Observation and Remark (O&R) forms. Note: during the early phase of purging, emphasis should be put on minimizing and stabilizing pumping stress, and recording those adjustments. Purging is considered complete and sampling may begin when all the above indicator field parameters have stabilized. Stabilization is considered to be achieved when three (3) consecutive readings, taken at three (3) to five (5) minute intervals, are within the following limits:

- Turbidity – 10% (if values are greater than 1 NTU)
- DO – 10%
- Specific conductance – 3%
- Temperature – 3%
- pH - +/- 0.1 units
- ORP - +/- 10 millivolts

Percentage difference should be calculated using the following formula:

(Absolute Value (Previous Value – Updated Value)/ Previous Value) * 100

All measurements should be obtained using a flow-through-cell. Transparent flow-through-cells are preferred, because they allow field personnel to watch for particulate build-up within the cell. This build-up may affect indicator field parameter values measured within the cell. If the cell needs to be cleaned during purging operations, continue pumping and disconnect cell for cleaning, then reconnect after cleaning and continue monitoring activities.

The flow-through-cell must be designed in a way that prevents air bubble entrapment in the cell. When the pump is turned off or cycling on/off (when using a bladder pump), water in the cell must not drain out. Monitoring probes must be submerged in the water at all times. If two flow-through-cells are used in series, the one with the dissolved oxygen probe should come first (this parameter is most susceptible to error if air leaks into the system).

4.3.5 Collect Water Sample

Water samples for laboratory analyses must be collected before water has passed through the flow-through-cell (use a by-pass assembly or disconnect cell to obtain the sample).

VOC samples should be collected first and directly into the sample containers. Sample water should be allowed to fill sample containers gently down the inside of the container with minimal turbulence.

During purging and sampling, the tubing should remain filled with water so as to minimize possible changes in water chemistry upon contact with the atmosphere. It is recommended that 1/4 inch or 3/8 inch (inside diameter) tubing be used to help insure that the tubing remains water filled. If the pump tubing is not completely filled to the sampling point, use one of the following procedures to collect samples: 1) add clamp, connector (Teflon or stainless steel) or valve to constrict sampling end of tubing; 2) insert small diameter Teflon tubing into water filled portion of pump tubing, allowing the end to protrude beyond the end of the pump tubing, collect samples from small diameter tubing; 3) collect non-VOC samples first, then increase flow rate slightly until the water completely fills the tubing, collect sample and record new drawdown, flow rate and new indicator parameter values. This third technique (least favorable because VOC samples should be collected first) allows the drawdown in the well to remain constant while all other samples are being collected. If flow is increased to collect VOC samples, do not allow the drawdown in the well to drop below the top of the well screen. Note that any two, or all three, techniques may be used in conjunction to achieve the desired results.

Add preservative, as required by analytical methods, to samples immediately after they are collected if the sample containers are not pre-preserved.

Label each sample as collected. Samples requiring cooling (VOCs, organics, cyanide etc.) will be placed in a cooler with ice or refrigerant for delivery to the laboratory. Metal samples after acidification to a pH less than 2 do not need to be cooled.

4.3.6 Posting Sampling Activites

After collection of the samples, the pump tubing may be dedicated to the well for re sampling (by hanging the tubing inside the well), decontaminated, or properly discarded.

Before securing the well, measure and record the well depth (to 0.01 feet), if not measured during the monthly water level measurements. Note: measurement of total well depth is optional after the initial low stress sampling event. However, it is recommended if the well has a "silting" problem or if confirmation of well identity is needed.

Secure the well.

4.4 Decontamination

Refer to SEI -5.1.5 *Maintenance and Decontamination of Field Equipment* for further information regarding the decontamination of field equipment. Decontamination procedures specific to low stress sampling are described below.

Decontaminate sampling equipment prior to use in the first well and following sampling of each subsequent well. The pump will not be removed between purging and sampling operations. The pump and tubing (including support cable and/or electrical wires which are in contact with the well and/or groundwater) will be decontaminated by the procedure described below.

4.4.1 Procedure

The decontaminating solutions can be pumped from either buckets or short PVC casing sections through the pump or the pump can be disassembled and flushed with decontaminating solutions. It is recommended that detergents and isopropyl alcohol be used sparingly in the decontamination process and water flushing steps be extended to ensure that any sediment trapped in the pump is removed. The pump exterior, safety cables, electrical wires and other re-usable parts must be rinsed with the decontaminating solutions, as well. The procedure is as follows.

1. Flush the equipment with potable water.
2. Flush with non-phosphate detergent solution. If the solution is recycled, the solution must be changed periodically.
3. Flush with potable or distilled/deionized water to remove all of the detergent solution. If the solution is recycled, the solution must be changed periodically.
4. Flush with methanol (pesticide grade). If equipment blank data from previous sampling event show that the level of contaminants is insignificant, then this step may be skipped.

Flush with analyte-free water. The final water rinse must not be re-used for additional decontamination procedures.

4.5 Quality Assurance/Quality Control

QA/QC samples (i.e. trip and equipment blanks and field duplicates) will be collected in accordance to the site Quality Assurance Project Plan, if appropriate or as specified in the site work plan.

Quality control samples are required to verify that the sample collection and handling process has not compromised the quality of the groundwater samples. All field quality control samples must be prepared the same as regular investigation samples with regard to sample volume, containers, and preservation.

Collect samples in order from wells with lowest contaminant concentration to the highest. Collect equipment blanks after sampling contaminated wells and not after background wells.

4.6 Documentation

A record of the sample collection must be made in the field at the time the sample is collected. The record should be made in a field notebook, a sample collection form, or other medium acceptable according to the study protocol. The information to be recorded must, at a minimum, include the following:

- Date and time of sample collection;
- Name(s) of the personnel performing the sampling;
- Weather;
- Sample location, well identification, and / or sample identification;
- Project/study designation;
- Water level prior to purging (equilibrium level);
- Total Well Depth;
- Well volume calculations;
- Volume purged from the well prior to sampling;
- Observations (I.E. PID reading or presence of NAPL); and
- Equilibrated chemical parameters monitored in the field

5.0 RESPONSIBILITIES

Stone staff are required to take accurate and descriptive notes. Variations from this SOP should be noted on observations and remarks (O&R) forms along with data and personnel.

Field staff members are responsible for following and implementing all procedures outline within this SOP. Care shall be taken to avoid compromising sample and data integrity.

6.0 DEFINITIONS

1. *pH-* The log-normal activity (concentration) of Hydrogen Ion (H^+) and Hydroxide Ion (OH^-) in solution. Ranges from 0 (acidic) to 7 (neutral) to 14 (basic).
2. *Conductivity-* The ability of water to conduct electricity as a function of dissolve ionic species, suspended solids, and organic content. Usually measured in Siemans (S).
3. *Turbidity-* The amount of suspended solids in a liquid.
4. *EPA-* The United States Environmental Protection Agency
5. *VOC-*Volatile Organic Compound.
6. *SVOC-*Semi-volatile Organic Compound.

7.0 REFERENCES

U.S. Environmental Protection Agency, Region 1, 1996, *Low Stress (low flow) Purging and Sampling Procedure for the Collection of Groundwater Samples from Monitoring Wells,* p-13

8.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

None

9.0 AUTHORIZATION

Revised by: _____ Date: 1/21/05

Michael D. Rossi, Senior Scientist

Approved by: _____ Date: 21 January 2005

Christopher T. Stone, President

10.0 REVISION HISTORY

None

STANDARD OPERATING PROCEDURE

SEI-6.39.1

Surface Sediment Sampling Using an EKMAN Bottom Grab Sampler

SOP Number: SEI-6.39.1
Revision Number: 1

Date Issued: 08/21/12
Date of Revision: 10/16/12

1.0 OBJECTIVE

This standard operating procedure (SOP) describes the procedures used to collect surface sediment with an EKMAN grab sampler. Surface sediment is typically analyzed for various physical and chemical variables. For the purposes of this SOP, surface sediment is defined as the upper 10 cm (4 inches) of the sediment column but may vary given the sampling interval specified in the study design.

A stainless-steel Ekman grab sampler is capable of collecting acceptable samples from a variety of soft substrates, such as silt, silt mixed with clay, and silt mixed with some sand. The EKMAN grab sampler has two doors on top to allow easy access to the sediment for visual characterization and sampling of surface sediments. The procedures for collecting surface sediment samples using the EKMAN grab sampler are described in Section 4.0.

2.0 POLICIES

1. Personnel will legibly record data and observations in the field to enable others to reconstruct project events and provide sufficient evidence of activities conducted.
2. Field personnel will use a field logbook, observation and remark form (O&R) or other designated field form to record activities, measurements, and observations made in the field.

3.0 SAFETY ISSUES

1. Whenever visiting a hazardous or potentially hazardous site, it is important to read and follow the site health and safety protocol (HASP).
2. Field visits may involve accessing remote areas. Health and safety concerns regarding these field visits are minimal however; slip, trip and fall hazards, poisonous plant dangerous animal, as well as getting vehicles stuck in remote areas do present considerable health and safety concerns. Collection of surface sediment samples may require accessing sample location using watercraft. All SEI employees must wear appropriate personal floatation devices if accessing work areas by watercraft. To help ensure field staff's health and safety with remote areas, all field staff are to have daily communication with the project manager or another appropriate SEI employee.

3. If necessary and appropriate, all chemicals are required to be received with a Material Safety Data Sheets (MSDS) or appropriate application label. These labels or MSDS shall be made available to all personnel involved in the sampling and testing.

4.0 PROCEDURES

4.1 Equipment and Materials

Equipment required for sediment sampling using the EKMAN grab sampler includes the following:

- Stainless-steel EKMAN Bottom grab sampler (typically 0.25 ft^2) with handle and rope
- Trigger weight (i.e., messenger)
- Teflon® or polyethylene tubing
- Flat-bottomed container (e.g., dish pan)
- Stainless-steel ruler
- Stainless-steel spoons
- Stainless-steel mixing bowl or pot
- Scrub brush
- Squirt bottles (for solvents)
- Alconox® (laboratory detergent)
- Acetone and hexane (if applicable for a specific project).

4.2 Procedure

4.2.1 Decontamination

Before each station is sampled, decontaminate the inner and outer surfaces of the grab sampler and all stainless-steel sample compositing equipment. Sediment sampling and compositing equipment will be decontaminated using the following general sequence: site water rinse, Alconox scrub and rinse, site water rinse, solvent rinse (if applicable for a specific project) with acetone and hexane (respectively), and a final site water rinse. Equipment used for compositing the sediment samples will follow the same basic decontamination sequence except that the final rinse will be with distilled/deionized water. If there is a significant lapse of time between decontamination of the sediment sampling and compositing equipment and collection of the sample, then the decontaminated sediment sampling and compositing equipment will be protected from additional contamination by wrapping it in foil (with the dull side of the foil touching the equipment) and placing it in clean bags for transport, if

necessary. All solvent rinsates will be collected and disposed of in accordance with state and federal regulations.

4.2.2 EKMAN Bottom Grab Sampler Deployment

1. **Depth of Sampling** will be project specific and pre-determined based on the waterbody and the study zone. For example: the bio active zone in a particular water body may be 15-20 centimeters below the top of sediment. Therefore, it is important to document the depth of sampling required for the project and document the study zone to ensure that the depth of the sampling is at the appropriate depth for the project.
2. If the water depth is less than 9 ft, attach the grab sampler to the metal handles. If the water depth is greater than 9 ft, use the rope to deploy the grab sampler.
3. Place the grab sampler on a decontaminated surface and open it.
4. Ensure that the two release wires are securely placed around the release pins.
5. Lower the sampler through the water column at a slow and steady speed.
6. Allow the grab sampler to contact the bottom gently, with only its weight being used to force it into the sediments. The sampler should never be allowed to "free fall" to the bottom because this may result in premature triggering, an excessive wake, or improper orientation upon contact with the bottom.
7. Deploy trigger weight (i.e., messenger) to release the doors on the bottom of the grab sampler.

4.2.3 EKMAN Bottom Grab Sampler Retrieval

1. After the grab sampler has rested on the bottom for approximately 5 seconds, begin retrieving it at a slow and steady rate.
2. After the grab sampler breaks the water surface, gently lower it into a clean, flat-bottomed container, while maintaining the grab sampler in an upright position.
3. Open the doors on the top of the grab sampler, and inspect the sample for acceptability. The following acceptability criteria should be satisfied:
    - The sampler is not overfilled with sample to the point that the sediment surface presses against the top of the sampler or is extruded through the top of the sampler.
    - Overlying water is present (indicating minimal leakage).
    - The overlying water is not excessively turbid (indicating minimal disturbance or winnowing).
    - The sediment surface is relatively undisturbed.
    - The desired penetration depth is achieved (see below).

If a sample fails to meet the above criteria, it will be rejected and discarded away from the station. Such disposal will comply with all federal, state, and local guidelines.

Penetration depth should be determined by placing a decontaminated stainless-steel ruler against the center of the inside edge of the opening on the top of one side of the grab sampler and extending it into the grab sampler until it is almost in contact with the top of the sample. The penetration depth is determined by the difference between that measurement and the total depth of the grab sampler.

4.2.4 Sample Removal and Processing

1. For acceptable samples, remove the overlying water by slowly siphoning it off near one or more sides of the grab sampler. Ensure that the siphon does not contact the sediments or that fine-grained suspended sediment is not siphoned off. If sediment is suspended in the overlying water, do not proceed with siphoning until the sediment is allowed sufficient time to settle.
2. After the overlying water is removed, characterize the sample as specified in the study design. Characteristics that are often recorded include but are not limited to:
   - Sediment type (e.g., silt, sand)
   - Texture (e.g., fine-grain, coarse, poorly sorted sand)
   - Color
   - Approximate percentage of moisture
   - Biological structures (e.g., chironomids, tubes, macrophytes)
   - Approximate percentage of biological structures
   - Presence of debris (e.g., twigs, leaves)
   - Approximate percentage of organic debris
   - Presence of shells
   - Approximate percentage of shells
   - Stratification, if any
   - Presence of a sheen
   - Odor (e.g., hydrogen sulfide, oil, creosote).
3. After the sample is characterized, remove the top 10 cm using a stainless steel spoon (see site-specific study design for project-specific sampling interval). Unrepresentative material (e.g., large shells, stones, leaves, twigs) should be carefully removed without touching the sediment sample under the supervision of the chief scientist and noted on the field logbook.
4. Remove subsamples for analysis of unstable constituents (e.g., volatile organic compounds, acid-volatile sulfides), and place them directly into sample containers without homogenization.
5. Transfer the remaining surface sediment to a stainless-steel mixing bowl or pot for homogenization. Additional grab samples may be required to collect the volume of

sediment specified in the study design. The mixing bowl should be covered with aluminum foil (dull side down) while additional grab samples are being collected to prevent sample contamination (e.g., from precipitation, splashing water, falling leaves).

6. After a sufficient volume of surface sediment from a grab is collected (i.e., 0−10 cm), move away from the sampling location, open the jaws of the grab sampler, and allow the remainder of the sediment sample to fall out of the grab sampler. Discard this material away from the station, and rinse away any sediment adhering to the inside of the grab sampler. The grab sampler is now ready for additional sampling at the same station or decontamination before sampling at a new station.
7. After a sufficient volume of sediment is transferred to the mixing bowl, homogenize the contents of the bowl using stainless-steel spoons until the texture and color of the sediment appears to be uniform.
8. After the sample is homogenized, distribute subsamples to the various containers specified in the study design and preserve the samples as specified in the study design.

4.3 Documentation

Label the sealed sample container with the following information (at a minimum):

1. Study or Project Number.
2. Date of sample collection.
3. Sample identification number (alpha-numeric label), if appropriate.
4. Depth interval.
5. Top of the interval.
6. Approximate amount of sample recovered.
7. Initials of Sampler
8. Other, as appropriate.

Document transfer of the samples to the laboratory or courier using appropriate chain of custody forms.

5.0 RESPONSIBILITIES

1. All personnel will legibly record data and observations (including phone conversations) in accordance with this SOP to enable others to reconstruct project events and provide sufficient evidence of activities conducted.

2. It is the responsibility of the employee to follow SOPs covering activities in his/her work area or identify a deviation from the written SOP in the field notes.

6.0 DEFINITIONS

1. *Observations & Remarks Form (O&R):* A pre-printed form, which contains mostly blank space for general note taking. The form typically prompts the user for the study or project designation, the SEI project number, the client or sponsor name, the total number of pages (page *n* of n) and requires a signature and date. The form is generally used to capture notes of one person when another, more specific forms is not available.

2. *Raw data* any laboratory worksheets, records, memoranda, notes, or exact copies thereof, that are the result of original observations and activities of a study and are necessary for the reconstruction and evaluation of the report of that study. In the event that exact transcripts of raw data have been prepared (e.g., tapes which have been transcribed verbatim, dated, and verified accurate by signature), the exact copy or exact transcript may be substituted for the original source as raw data. Raw data may include photographs, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments.

7.0 REFERENCES

40 CFR Part 160 Good Laboratory Practice Standards, August, 1989.

8.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

None

9.0 AUTHORIZATION

Written by: Lee Rosberg Date: 10/16/12

Lee J. Rosberg, Project Scientist

Approved by: [signature] Date: 10/16/12

Christopher T, Stone, President

10.0 REVISION HISTORY

Revision 1

1. Section 4.2.3 Added disposal of unwanted sample will comply with all federal, state, and local guidelines.
2. Section 4.2.2 Added depth of sampling and need to know the depth of sampling prior to deployment to ensure that the depth of the sampler is appropriate for the study zone.

STANDARD OPERATING PROCEDURE

SEI-6.40.0

Sediment Porewater Sampling using a (Henry's) Push Point (PP) Sampler

SOP Number: SEI-6.40.0
Revision Number: 0

Date Issued: 8/21/12
Date of Revision: NA

1.0 OBJECTIVE

The objective of this Standard Operating Procedure (SOP) is to provide a standardized method for collection of sediment porewater samples from a micro (Henry's) Push Point sampler or mini piezometers. Sampling locations must be chosen in line with the objectives of the data required, with care taken to ensure sample integrity.

2.0 POLICIES

1. Personnel will legibly record data and observations in the field to enable others to reconstruct project events and provide sufficient evidence of activities conducted.
2. Field personnel will use a field logbook, observation and remark form (O&R) or other designated field form to record activities, measurements, and observations made in the field.

3.0 SAFETY ISSUES

1. Whenever visiting a hazardous or potentially hazardous site, it is important to read, follow and sign the site health and safety protocol (HASP).
2. Field visits may involve accessing remote areas. Health and safety concerns regarding these field visits are minimal however; slip, trip and fall hazards, poisonous plant dangerous animal, as well as getting vehicles stuck in remote areas do present considerable health and safety concerns. Collection of surface sediment samples may require accessing sample location using watercraft. All SEI employees must wear appropriate personal floatation devices if accessing work areas by watercraft. To help ensure field staff's health and safety with remote areas, all field staff are to have daily communication with the project manager or another appropriate SEI employee.
3. Health and safety of the sampling personnel is of outmost importance. Field personnel will use the "buddy system" when working within fast-moving streams.

4.0 PROCEDURES

4.1 Equipment and Materials

1. Micro Push Point (PP) Samplers - in lengths of 14" and 27" (referred to as PP14 and PP27).
2. Syringe Assemblies - 50 ml, 100% polyethylene/polypropylene syringes configured to tygon tubing (1/4" OD x 1/8" ID) with clamps (to secure tubing to sampling port) and a stopper.
3. Peristaltic Pump - either battery-powered or with AC car-adapter unit.
4. Collection Containers and Preservative - as specified in the site specific Quality Assurance Project Plan (QAPP) and Field Sampling Plan (FSP).
5. Decontamination Equipment - used to backflush cleaning solution through the PP samplers for decontamination.
6. Gloves - for personal protection and to prevent cross-contamination of samples. May be plastic or latex; should be disposable and powderless.
7. Sampling Flags - used for identifying porewater sampling locations.
8. Field Notebook - a bound book used to record progress of sampling effort and record any problems and field observations during sampling.
9. Permanent Marking Pen - used to identify sample containers and for documentation of field logbooks and data sheets.
10. Cleaning Solution - used to decontaminate the PP samplers after use.
11. Deionized Water - used to rinse cleaning solution from the PP samplers during decontamination.
12. Trash Bag - used to dispose of gloves and any other non-hazardous waste generated during sampling and decontamination.
13. Plastic Waste Bottle - used to dispose of decontamination waste.

4.2 Porewater Sample Collection

Push point samplers consist of two pieces: a guard rod and a sampler (see Figure 1). The sampler is a rigid 1/8-inch diameter probe made of 316 stainless steel. The sampler has a short screened zone at one end (with approximately 20% open/screened area) and a sample port at the other end. The guard rod is inserted into the sampler body to provide support and prevent plugging during insertion into sediments. A syringe or tygon tubing is attached to the sample port for measurement of head and/or collection of a porewater sample. Push point samplers are available in various lengths. Water depth at the sampling location and the desired depth of sample collection determine the necessary length of the sampler.

4.2.1 Preparation for Sample Collection

The sampling team will wade to the specified sampling location and mark the location with a pin flag on the shore. The sample location (and GPS coordinates if required) will be recorded prior to collecting the sediment porewater sample. Insert the guard-rod into the mini piezometer body so that the guard rod and handle are squeezed together (Figure 2). Holding the device in this manner, push the push point sampler into the sediment to the desired depth using a gentle twisting motion. When the desired depth or refusal is reached, remove the guard rod from the body without disturbing the

position of the deployed sampler. The sampler shall be inserted approximately 1-2 feet below the sediment surface to refusal. In no case shall the sampler be entirely immersed in water.

After reaching the desired depth, remove the guard rod from the Push Point Sampler. Once the guard rod has been removed it SHOULD NOT be reinserted into the device until the bore holes are thoroughly cleansed of all sand, silt and other debris. In addition, once the guard rod has been removed from the sampler, the sampler should not be pushed further into the sediments. This may damage the screened zone and/or plug the sampler with sediment.

Using the meter stick record the depth of the water (distance from the sediment top to the top of the surface water) and the length of the sampler that is not immersed in the sediment (distance from the sediment to the top of the sampler). Record this information in the field log book.

4.2.2 Collection of Groundwater Flux Measurements

After insertion into the sediment, the sampler can then be used as a mini-piezometer to determine the potential direction of groundwater flux. Connect a piece of tygon tubing to the sample port (see Figure 3). Be sure to place a clamp at the mouth of the sampler tubing to ensure a good seal at the sampling port of the sampler.

Pump out any remaining air in the sampler and/or tubing by extending the 50 ml syringe and inserting it into the open end of the tygon tubing. Squeeze the syringe to blow any air and/or water out of the tubing and the micro Push Point Sampler. Slowly remove the syringe from the Taigon Tubing and hold the tubing as diagramed in Figure 3. Allow the water to rise to its static level. When the water level appears to have stabilized, record on the field data sheet a "positive" or "negative" groundwater flux observation at the respective sampling location. Using the meter stick, measure the distance between the water level in the tubing and the surface water and record this measurement on the field data sheet.

A positive flux measurement indicates that the groundwater is moving towards the surface water body, or the surface water body is "gaining" groundwater. A negative flux measurement indicates that the groundwater is moving away from the surface water body, or the surface water body is "losing" water to the groundwater.

4.2.3 Collection of Sediment Porewater Samples for Analysis

Attach a "syringe assembly" (a pre-assembled 50 ml, 100% polyethylene/polypropylene syringe clamped to Taigon tubing) or peristaltic pump to the sampler port using a length of Taigon tubing. Be sure to place a clamp at the mouth of the sampler tubing to ensure a good seal at the sampling port. Withdraw water at a low-flow sampling rate (50-200 ml/min) until extracted water becomes non-turbid. Withdraw adequate amounts of porewater and transfer the sample into a labeled sample collection container as specified in the QAPP.

If sampling equipment is to be re-used, follow the appropriate decontamination procedures before collecting the next sample.

4.3 Sample Preservation, Containers, Handling, and Storage

Once samples have been collected, the following procedures should be followed:

1. Transfer the sample(s) into suitable, labeled sample containers specific for the analyses to be performed.
2. Preserve the sample, if appropriate, or use pre-preserved sample bottles. Do not overfill bottles if they are pre-preserved.
3. Cap the container securely, place in a resealable plastic bag, and cool to 4°C on wet ice.
4. Record all pertinent data in the site logbook and/or on field data sheets.
5. Complete the Chain of Custody record.
6. Attach custody seals to cooler prior to shipment.
7. Decontaminate all non-dedicated sampling equipment prior to the collection of additional samples.

4.4 Interferences and Potential Problems

Care should be taken to avoid tracking sediment and/or silt from one area to another. As samples are taken sequentially, care should also be taken not to contaminate an area yet to be sampled with the residue of the sample that is currently being taken. In general one should move in a single direction through the sampling area. If an area is known or suspected of having a higher concentration of contaminants, all other considerations being equal, it should be sampled last to prevent cross contamination.

There are two primary interferences or potential problems associated with sediment porewater sampling. These include cross contamination of samples and improper sample collection.

1. Cross contamination problems can be eliminated or minimized through the use of dedicated or disposable sampling equipment. If this is not possible or practical, then decontamination of sampling equipment is necessary.

2. Improper sample collection can involve using contaminated equipment, equipment that is potentially not compatible with the contaminants of concern, disturbance of the stream or impoundment substrate, and sampling in an obviously disturbed or non-representative area. Be sure to use sampling equipment of an appropriate composition based upon the suspected contaminants and analyses to be performed.

Following proper decontamination procedures, minimizing disturbance of the sample site, and careful selection of sampling locations will eliminate these problems. Proper timing for the collection of samples must be taken into consideration due to tidal influences and low or fast flowing streams or rivers.

4.5 Record keeping and Quality Control

There are no specific quality assurance activities which apply to the implementation of these procedures. However, the following general procedures apply:

1. All data must be documented on field data sheets or within site logbooks.
2. All instrumentation must be operated in accordance with operating instructions as supplied by the manufacturer, unless otherwise specified in the work plan. Equipment calibration activities must occur prior to sampling/operation and they must be documented.

Descriptions of any deviations and the reason for deviations from the site QAPP or this SOP should be noted in the field notebook, as necessary. In addition, the logbook should track pertinent sample collection information such as: sample date/time, personnel, weather conditions, and sample identification information. Samples taken from areas with visible staining or other indications of non-homogeneous conditions should be noted. Field personnel will collect the proper type and quantity of quality control samples as prescribed in the QAPP.

4.6 Decontamination

Because decontamination procedures are time-consuming, having a quantity of sampling tools sufficient to require decontamination at a maximum of once per day is recommended. All sampling equipment must be decontaminated prior to reuse. Equipment decontamination will consist of the following 4 steps:

1. Cleaning Solution
2. Deionized water rinse
3. Acetone rinse
4. Deionized water rinse

Begin decontamination of the push point sampler by thoroughly removing all sand, silt etc. from the guard rod and the exterior of the sampler. Prepare three dedicated decontamination syringes with cleaning solution, deionized water, and acetone, respectively. Connect the cleaning syringe filled with cleaning solution to the end of the sampler port. Push the contents of the cleaning syringe through the sampler into a waste receptacle. Gently push the guard rod all the way into the bore of the Push Point sampler to dislodge any bridged material. Re-rinse the Push Point sampler with cleaning solution. Follow this with a distilled water/and or acetone rinse. Rinse the guard rod with cleaning solution, followed with a distilled water rinse then and acetone rinse followed by a second distilled water rinse. Reinsert the guard-rod into the push point sampler and the device is ready for re-use.

Note: Before the guard-rod is reinserted into the push point sampler, all small bends in both the guard-rod and in the sampler should be removed. Use caution when straightening the screened-zone, it is somewhat delicate without the guard-rod inside it, and can be broken through repeated bending. It is sometimes helpful when straightening the screened zone to insert the guard rod or the cleaning rod to the area of the bend in the screened zone. Gently unbend the portion of the screened zone nearest the rod and carefully advance the rod to the next bend. After the rod has been fully inserted into the screened zone perform the final screened zone straightening fine-tuning until the guard rod slides freely through it.

All marker flags (if reused) should be decontaminated by wiping off with towels and/or baby wipes before re-use.

4.7 Site Clean-Up

Disposable personal protective equipment and other non-hazardous waste generated during sampling and decontamination activities will be placed in a trash bag and taken to a waste receptacle at the field office to prevent disturbance by animals and dispersion by wind. All non-hazardous waste will be disposed of in municipal waste bins.

5.0 RESPONSIBILITIES

1. All personnel will legibly record data and observations (including phone conversations) in accordance with this SOP to enable others to reconstruct project events and provide sufficient evidence of activities conducted.
2. It is the responsibility of the employee to follow SOPs covering activities in his/her work area or identify a deviation from the written SOP in the field notes.

6.0 DEFINITIONS

1. *Observations & Remarks Form (O&R):* A pre-printed form, which contains mostly blank space for general note taking. The form typically prompts the user for the study or project designation, the Stone project number, the client or sponsor name, the total number of pages (page *n* of n) and requires a signature and date. The form is generally used to capture notes of one person when another, more specific forms is not available.
2. *Raw data* any laboratory worksheets, records, memoranda, notes, or exact copies thereof, that are the result of original observations and activities of a study and are necessary for the reconstruction and evaluation of the report of that study. In the event that exact transcripts of raw data have been prepared (e.g., tapes which have been transcribed verbatim, dated, and verified accurate by signature), the exact copy or exact transcript may be substituted for the original source as raw data. Raw data may include photographs, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments.

7.0 REFERENCES

40 CFR Part 160 Good Laboratory Practice Standards, August, 1989.

MHE Products. 2003. Operators Manual and Applications Guide. Models PP27, PP14, PPX36, and PPX72. Version 2.01. February 15, 2003.

8.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

Figure 1: Disassembled and Assembled Sampler Diagram

Figure 2: Guard Rod Inserted into Mini Piezometer Body Diagram

Figure 3: Collection of Groundwater Flux Measurements Diagram

9.0 AUTHORIZATION

Written by: Lee Rosberg Date: 8/23/12

Lee J. Rosberg, Project Scientist

Approved by: David J. Healy Date: 8/23/12

David J. Healy, Vice President

10.0 REVISION HISTORY

Not Applicable

Figure 1: Disassembled and Assembled Sampler Diagram

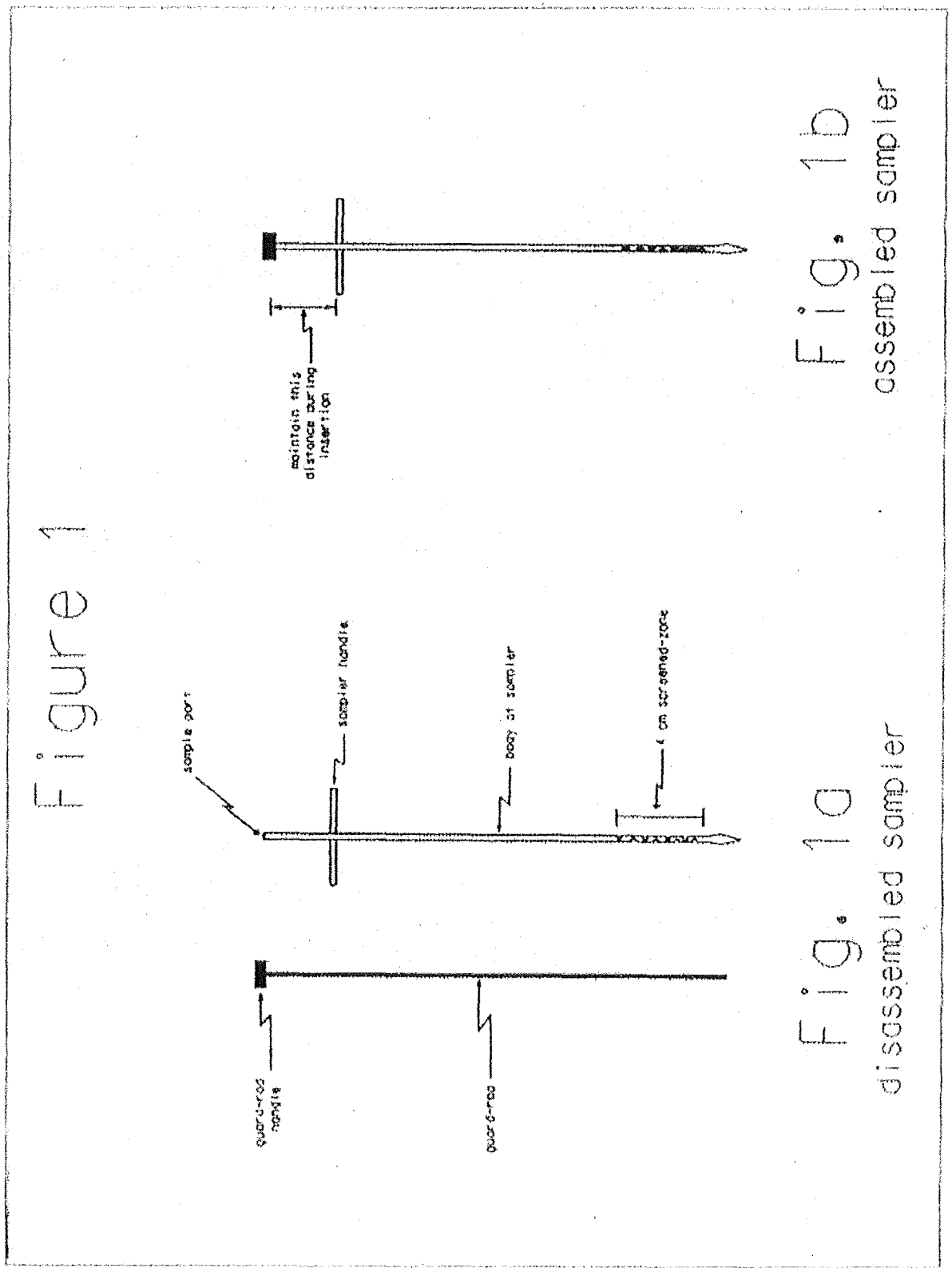


Figure 2: Guard Rod Inserted into Mini Piezometer Body Diagram

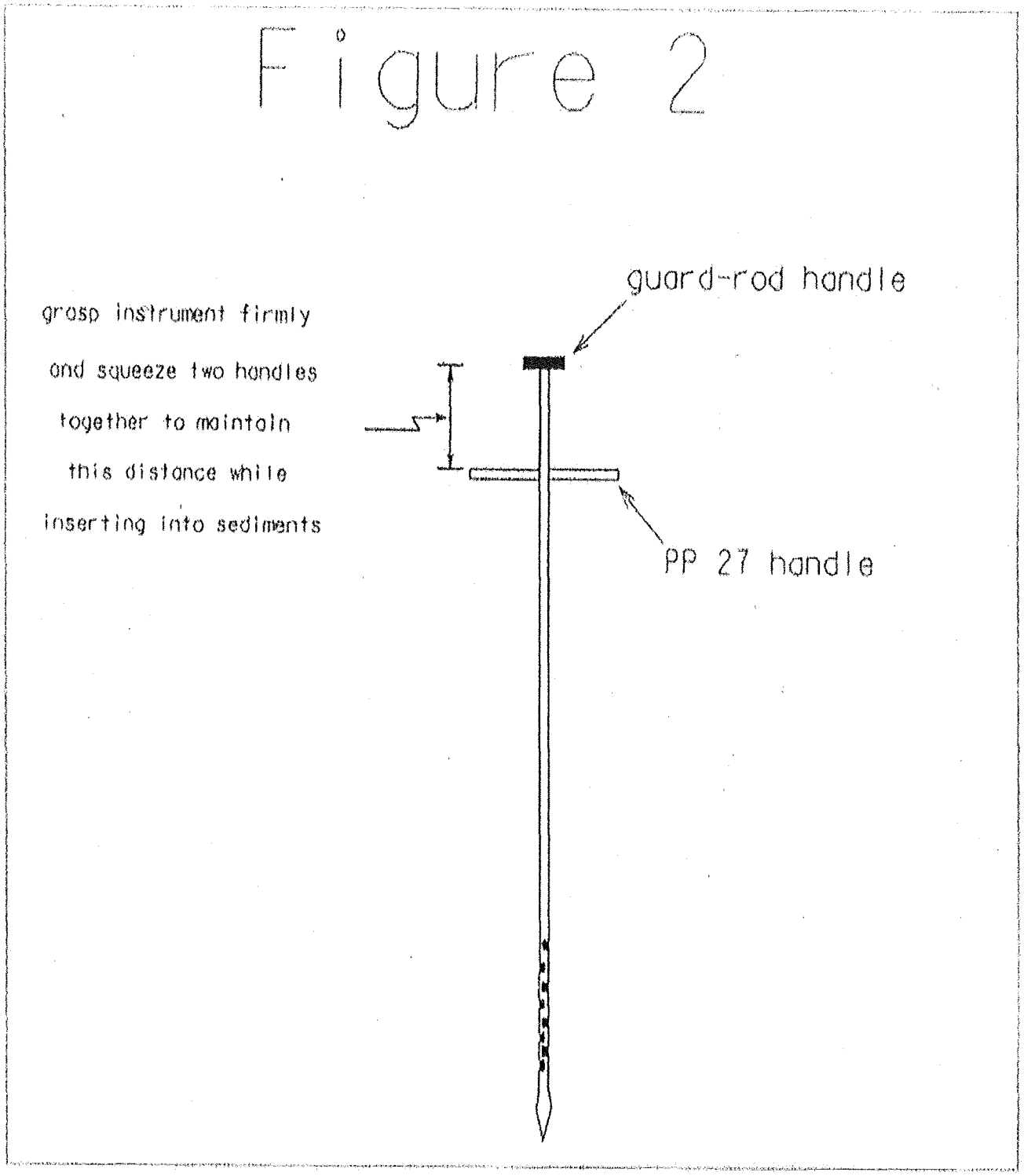
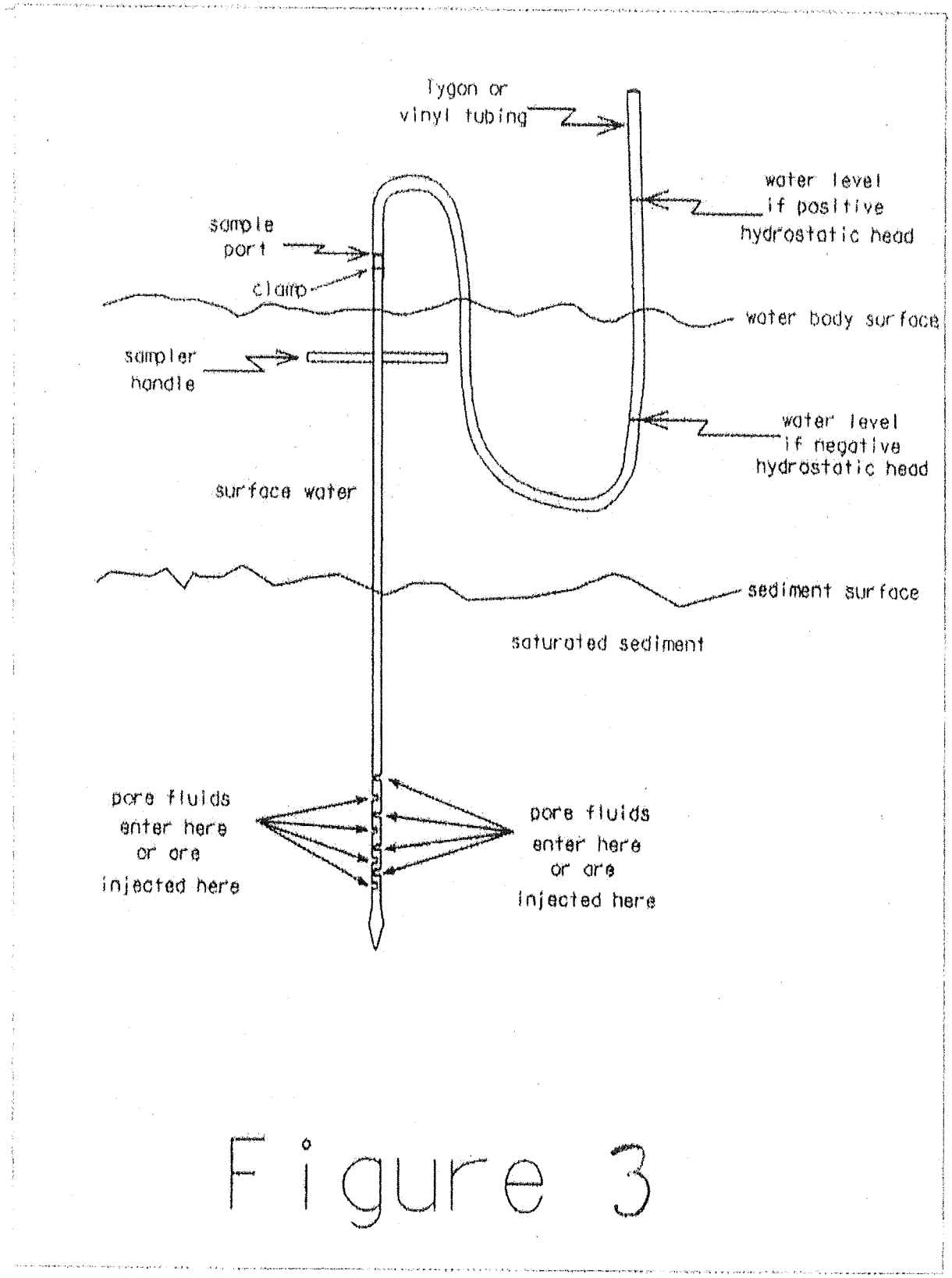


Figure 3: Collection of Groundwater Flux Measurements Diagram



STANDARD OPERATING PROCEDURE

SEI-6.41.0

Hydrophobic Dye Testing for Light or Dense Non-Aqueous Phase Liquids (NAPL) Contamination

SOP Number: SEI-6.41.0
Revision Number: 0

Date Issued: 08/21/12
Date of Revision: NA

1.0 OBJECTIVE

The objective of this standard operation procedure (SOP) is to describe the procedures used to collect soil and rock core samples for hydrophobic dye testing. Hydrophobic dye testing is used to determine the presence of non-aqueous phase liquids (NAPLs) in ex-situ soil samples suspected of NAPL contamination based on varying lines of evidence such as olfactory, visual, Photo-ionization detector (PID) screening, membrane interface probe (MIP) results, etc. that indicate the presence of high concentrations of total volatile organic compounds (VOCs) in subsurface soils or rock cores. Hydrophobic dye testing can be conducted on soils or rock core suspected of either light or dense NAPL contamination (LNAPL and DNAPL, respectively). Hydrophobic dyes (e.g. Sudan IV and Oil Red O) only test whether or not NAPLs are present in soil and are designed to work with other analytical technologies.

2.0 POLICIES

1. According to 40 CFR Part 160, Subpart E, Section 160.81, a testing facility shall have standard operating procedures in writing, setting forth study methods that management is satisfied are adequate to ensure the quality and integrity of the data generated in the course of a study.
2. Personnel will legibly record data and observations in the field to enable others to reconstruct project events and provide sufficient evidence of activities conducted.

3.0 SAFETY ISSUES

1. If necessary and appropriate, a site-specific health and safety plan shall be created for each study site. A template for creating a proper health and safety plan is provided on the SEI network.
2. Care must always be taken when approaching a sampling location. Do not, under any circumstances, place yourself in danger to collect a sample.
3. If necessary and appropriate, all chemicals are required to be received with Material Safety Data Sheets (MSDS) or appropriate application labels. These labels or MSDS shall be made available to all personnel involved in the sampling and testing.

4. Sudan IV is classified as a category 3 carcinogen and should be handled with extreme care. Stone personnel should not prepare hydrophobic dye test jars with Sudan IV, these tests kits can be purchased from vendors such as Pine Environmental under the trade name Oil-In-Soil, which contain a de minimis amount of Sudan IV. Oil Red O is hazardous in case of ingestion and slightly hazardous in case of skin contact, eye contact, and inhalation but in general is much less toxic to human health than Sudan IV and is the preferred hydrophobic dye for Stone personnel to use for hydrophobic dye testing.

4.0 PROCEDURES

4.1 Equipment and Materials

1. Hydrophobic Dye (Sudan IV or Oil Red O)
2. Stainless steel scoopula
3. Clean 4 oz. jars
4. Deionized or distilled water
5. Hammer and rock chisel
6. Protective gloves and safety glasses
7. Alconox and container for decontamination fluids
8. Stainless steel trimming cell (for rock core samples)

4.2 Sample Container Preparation

Certified clean 4 oz. soil sampling jars shall be procured from a laboratory or glassware vendor. The number of jars to be prepared is contingent on the scope and objectives of each individual study. A small amount (<1g) of Oil Red O is placed into each jar taking care to avoid skin contact or inhalation using a clean scoopula. Deionized water is added to each jar so that the jar is approximately half full. Jars are then placed into boxes or packaged in a manner that will prevent breakage when transported to the study site.

4.3 Hydrophobic Dye Testing for Soil Cores

The number of hydrophobic dye tests to be performed depends on the data objectives of ach specific study. When a soil core is retrieved that is potentially contaminated with NAPLs identify any intervals that most likely contain the NAPLs. This is usually accomplished by visual and olfactory observation and by screening the oil core with a PID. Headspace readings that are greater than 100 ppm have been suggested to indicate the potential for NAPLs (Ciba 1998: Watkins et al., 1995).

Once an interval is selected for hydrophobic dye testing don clean nitrile gloves and using a clean stainless steel scoopula carefully remove approximately 10g of soil from that interval, taking care to exclude liquids and soils that may have smeared along the core liner, and place it in an unused jar containing the hydrophobic dye and deionized water and shake vigorously for a few seconds. A bright red coloration appears in the presence of NAPLs. Record the results of the hydrophobic dye test on appropriate field forms. This color change confirms the presence of NAPLs; however conversely, no color change is not necessarily proof there is no NAPLs in the soil sample or the soil horizon from which it was taken. Further chemical analysis can provide the data necessary to calculate the potential presence of NAPLs.

Decontaminate scoopulas according to study specific decontamination protocols.

4.4 Hydrophobic Dye Testing for Rock Cores

Hydrophobic dye testing for rock cores is performed in the same manner as described for soil cores with the exception of core preparation. Once a rock core interval is selected for testing remove an approximately one inch thick section of the core using a clean hammer and chisel, be sure to wear safety glasses. Place the section of core on a clean surface, such as a stainless steel trimming cell, and trim the outer portions of the core using the hammer and chisel. Place the inner portion of the core subsample in the jar prepared with Oil Red O and deionized water and shake vigorously. Record the results of the test on appropriate field forms.

Decontaminate chisel and trimming cell according to study specific protocols.

5.0 RESPONSIBILITIES

1. All personnel will legibly record data and observations (including phone conversations) in accordance with this SOP to enable others to reconstruct project events and provide sufficient evidence of activities conducted.

2. It is the responsibility of the employee to follow SOPs covering activities in his/her work area or identify a deviation from the written SOP in the field notes

6.0 DEFINITIONS

1. *Observations & Remarks Form (O&R):* A pre-printed form, which contains mostly blank space for general note taking. The form typically prompts the user for the study or project designation, the Stone project number, the client or sponsor name, the total number of pages (page *n* of n) and requires a signature and date. The form is generally used to capture notes of one person when another, more specific forms is not available.

2. *Raw data* any laboratory worksheets, records, memoranda, notes, or exact copies thereof, that are the result of original observations and activities of a study and are necessary for the reconstruction and evaluation of the report of that study. In the event that exact transcripts of raw data have been prepared (e.g., tapes which have been transcribed verbatim, dated, and verified accurate by signature), the exact copy or exact transcript may be substituted for the original source as raw data. Raw data may include photographs, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments.

7.0 REFERENCES

40 CFR Part 160 Good Laboratory Practice Standards, August, 1989.

Ciba Specialty Chemicals. 1998. Final Non-Aqueous Phase Liquid (NAPL) Action Plan Investigation Report, Toms River Site. Ciba Specialty Chemicals Corporate Remediation, Toms River, New Jersey.

Watkins, M., E. Sieber, and G. Christians. 1995. An integrated approach for assessing the potential presence and distribution of DNAPLs at a Superfund site in New Jersey. Proceedings of the Ninth National Outdoor Action Conference, National Ground Water Association.

8.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

None

9.0 AUTHORIZATION

Written by: Lee Rosberg Date: 8/23/12

Lee Rosberg, Project Scientist

Approved by: David J. Healy Date: 8/23/12

David J. Healy, Vice President

10.0 REVISION HISTORY

Not Applicable

STANDARD OPERATING PROCEDURE

SEI-6.44.0

PROCEDURE FOR SAMPLE COLLECTION from DRINKING WATER SUPPLIES

SOP Number: SEI-6.44.0 Date Issued: 08/27/12
Revision Number: NA Date of Revision: NA

1.0 OBJECTIVE

This Standard Operating Procedure (SOP) provides a framework for sample collection from drinking water supplies. Drinking water supplies from residences or commercial establishments are often sampled during the course of a hydrogeological investigation. The principle reason for obtaining water samples from a drinking water supply is to learn more about the quality of water available for consumption or use.

Drinking water is most often obtained from one or more of three types of sources: wells completed in bedrock aquifers, wells or springs completed in surficial materials, or surface water. Drinking water supplies can be sampled to detect the presence of chemicals, ions or organisms in the supply. Samples can also be taken to measure the composite quality of the water that the residents are consuming, including any impact that the water delivery system might have. The exact number, type, location and analytical method for samples are highly dependent upon the objectives of the investigation.

Since drinking water supplies are usually in use and privately owned, sampling is performed at the owner's convenience. Thus, planning for the sampling event is a key component of the SOP.

2.0 POLICIES

It is the policy of Stone Environmental Inc. (Stone) that all field staff conducting this sampling read this SOP prior to acquiring samples. Refer to SEI 6.16.4 for sample handling, preservation and transportation procedures.

Stone personnel will legibly record data and observations in the field to enable others to reconstruct project events and provide sufficient evidence of activities conducted. Field personnel will use a field logbook, observation and remark form or other designated field form to record activities, measurements, and observations made in the field.

3.0 SAFETY ISSUES

1. Whenever visiting a hazardous or potentially hazardous site, it is important to read, follow and sign the site health and safety protocol (HASP).

2. Field visits may involve accessing remote areas. Health and safety concerns regarding these field visits are minimal however; slip, trip and fall hazards, and poisonous plant and dangerous animal, as well as getting vehicles stuck in remote areas do present considerable health and safety issues. To help ensure field staff's health and safety with remote areas, all field staff is to have daily communication with the project manager or another appropriate Stone Environmental, Inc. (Stone) employee.
3. Whenever sampling residential water supply wells the "buddy system" should be employed to minimize any risk of harm to Stone employees.

4.0 PROCEDURES

4.1 Planning

Drinking water sampling events must be well-planned and scheduled so that all locations are accurately sampled and documented. The actual field work involved in sampling drinking water supplies will be minimized if the event is well-planned. Planning efforts should cover at least the following topics:

1. Availability: Plan the sampling event for a date and time that is convenient for the owner of the supply. Make an appointment for each supply but do not overschedule. Keep all appointments or reschedule in advance.

2. Sample locations: The locations should be selected in advance according to the objectives of the investigation. Make a chart of locations and state the rationale for sampling those locations. The chart should be placed in the project file.

3. Analytical parameters: The parameters to be sampled for should be selected in advance according to the objectives of the study. Sample bottles should be acquired and labeled prior to fieldwork. Preservatives can be added either in advance or after the sample has been collected. The types of bottles and preservatives should be specified in accord with the project specific work plan.

4. Sample collection stations: The project work plan should specify the preferred sampling station at each sampling location. The two common stations are point of consumption samples such as kitchen taps, or point of entry samples, such as at pressure tanks or wells. Intermediate points are sometimes sampled to isolate sources of influence. The exact station will depend on whether the investigation focuses on the influence of the water delivery system on water quality.

4.2 Fieldwork

Samples of drinking water are obtained in the following manner:

1. Obtain access to the specified sampling location.

2. Open the sampling port. For interior ports this may require the use of a hose or bucket to remove purge water. Remove aerators from taps.

3. Allow the water to run freely until distribution system is flushed. This is most conveniently determined by monitoring temperature of the water. The system is flushed for 10 minutes or until the temperature of the water stabilizes, whichever is later. If required by a site specific work plan monitor physical/chemical parameters (turbidity, specific conductance, pH, ORP, DO) every three (3) to five (5) minutes. Values are to be recorded on appropriate field forms or on Observation and Remark (O&R) forms. Purging is considered complete and sampling may begin when all the above indicator field parameters have stabilized. Stabilization is considered to be achieved when three (3) consecutive readings, taken at three (3) to five (5) minute intervals, are within the following limits:

    - Turbidity – 10% (if values are greater than 1 NTU)
    - DO – 10%
    - Specific conductance – 3%
    - Temperature – 3%
    - pH - +/- 0.1 units
    - ORP - +/- 10 millivolts

    Percentage difference should be calculated using the following formula:

    (Absolute Value (Previous Value – Updated Value)/ Previous Value) * 100

    All measurements should be obtained using a flow-through-cell. Transparent flow-through-cells are preferred, because they allow field personnel to watch for particulate build-up within the cell. This build-up may affect indicator field parameter values measured within the cell. If the cell needs to be cleaned during purging operations, continue pumping and disconnect cell for cleaning, then reconnect after cleaning and continue monitoring activities.

    The flow-through-cell must be designed in a way that prevents air bubble entrapment in the cell. Monitoring probes must be submerged in the water at all times. If two flow-through-cells are used in series, the one with the dissolved oxygen probe should come first (this parameter is most susceptible to error if air leaks into the system).

4. When ready to sample allow water to fill sampling containers, add preservative as required by analytical methods, then cap and label all samples. Place samples in a cooler chilled to 4ºC until delivered to the analytical laboratory.

   Water samples for laboratory analyses must be collected before water has passed through the flow-through-cell (use a by-pass assembly or disconnect cell to obtain the sample).

   VOC samples should be collected first and directly into the sample containers. Sample water should be allowed to fill sample containers gently down the inside of the container with minimal turbulence.

5. Document the collection of all samples in the field notebook (SEI SOP 4.5.10). Include the following information: location, ate, time, exact sample station, time flushed, stabilization temperature, number of samples taken and ay unusual observations or events.

4.3 Follow-Up

The owner of a home or business rarely receives a request for permission to obtain water samples. Thus, the act of sampling raises some questions for most owners. A brief yet comprehensive follow-up to the sampling event, along with a contact phone number for more information, is critical for maintaining good will and access to that sampling location. A short letter of transmission with a brief description of the results and their comparison with the drinking water standards is an effective tool for samplers. Do not transmit unexplained water quality results to the homeowner as most will not have a background in analysis of test results. Any follow-up letter should be written in clear, unambiguous terms.

4.4 Quality Assurance/Quality Control

QA/QC samples (i.e. trip and equipment blanks and field duplicates) will be collected in accordance to the site Quality Assurance Project Plan, if appropriate or as specified in the site work plan.

Quality control samples are required to verify that the sample collection and handling process has not compromised the quality of the water supply samples. All field quality control samples must be prepared the same as regular investigation samples with regard to sample volume, containers, and preservation.

5.0 RESPONSIBILITIES

Stone staff are required to take accurate and descriptive notes. Variations from this SOP should be noted on observations and remarks (O&R) forms along with data and personnel.

Field staff members are responsible for following and implementing all procedures outline within this SOP. Care shall be taken to avoid compromising sample and data integrity.

6.0 DEFINITIONS

1. *pH*- The log-normal activity (concentration) of Hydrogen Ion (H^+) and Hydroxide Ion (OH^-) in solution. Ranges from 0 (acidic) to 7 (neutral) to 14 (basic).
2. *Conductivity*- The ability of water to conduct electricity as a function of dissolve ionic species, suspended solids, and organic content. Usually measured in Siemans (S).
3. *DO*- Dissolved oxygen- The amount of oxygen dissolved in a body of water at a given temperature and atmospheric pressure. It is usually expressed as a concentration in mg/L or as a percentage of saturation.
4. *ORP*- Oxidation Reduction Potential- Oxidation is a term used to denote the occurrence of a molecule losing an electron. Reduction occurs as a molecule gains an electron. The "potential is an indication of a solution's propensity to contribute or accept electrons.
5. *Turbidity*- The amount of suspended solids in a liquid.
6. *VOC*-Volatile Organic Compound.

7.0 REFERENCES

None

8.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

None

9.0 AUTHORIZATION

Written by:_____ Date:_____

Lee J. Rosberg, Project Scientist

Approved by:_____ Date:_____

Christopher T. Stone, President

10.0 REVISION HISTORY

None

STANDARD OPERATING PROCEDURE

SEI-6.45.0

Collection, Handling, and Preservation of Discrete Soil Samples

SOP Number: SEI-6.45.0
Revision Number: 0

Date Issued: 08/27/12
Date of Revision: NA

1.0 OBJECTIVE

The purpose of this procedure is to provide instruction for the collection, handling, and preservation of discrete soils for various analyses including, but not limited to: the potential presence of Volatile Organic Compounds (VOCs) along with representative samples for physical property analysis including porosity, bulk density, and fraction of organic carbon content (*foc*), Semi-Volatile Organic Compounds (SVOCs), Polycyclic Aromatic Hydrocarbons (PAHs), Metals, and Polychlorinated Biphenyls (PCBs).

VOC samples need to be obtained, logged, and preserved into methanol with a minimal loss of volatile analytes.

2.0 POLICIES

1. According to 40 CFR Part 160, Subpart E, Section 160.81 and the NELAC standards, a testing facility shall have standard operating procedures in writing setting forth study methods that management is satisfied are adequate to insure the quality and integrity of the data generated in the course of a study.
2. Personnel will legibly record data and observations in the field to enable others to reconstruct project events and provide sufficient evidence of activities conducted.

3.0 SAFETY ISSUES

1. If necessary and appropriate, a site-specific health and safety plan shall be created for each study site. A template for creating a proper health and safety plan is provided on the SEI network.
2. Care must always be taken when approaching a sampling location. Do not, under any circumstances, place yourself in danger to collect a sample.
3. If necessary and appropriate, all chemicals are required to be received with Material Safety Data Sheets (MSDS) or appropriate application label. These labels or MSDS shall be made available to all personnel involved in the sampling and testing.

4.0 PROCEDURES

4.1 Equipment and Materials

1. For VOCs: Pre-weighed VOA vials with screw cap and silicone septum containing 10ml of purge-and-trap grade methanol (MSDS, Attachment 1). Appropriate sample containers as needed for other analytes

2. For VOCs: Sample collection device for minimally disturbed delivery of sample such as:
   - Stainless steel or disposable plastic syringe with barrel cut-off and smaller than vial opening
   - Stone's custom made stainless steel soil samplers with removable plungers
   - Encore piston sampler

3. Stainless steel spoons and bowls

4. Hand auger or other manual soil sampling tool

5. Portable balance, accurate to 0.01 grams, and calibration weights for vial preparation

6. Decontamination supplies if reusing stainless steel sampling tools: distilled water, alconox, scrub brushes, buckets, methanol if specified in project work plan

7. Field notebook, sample collection form, observation and remark form, or other acceptable medium for recording field notes. Ballpoint, indelible pens

8. Portable photoionization detector (PID) with calibration gas

9. Hacksaw

10. Soil core caps of appropriate diameter for the drill tooling being used to retrieve cores

11. Plastic wrap and zip top bags

12. Shipping materials: coolers with ice, bubble wrap or other packaging materials, packing tape

13. Measuring tape

14. As necessary or as described in the Site HASP appropriate personnel protection equipment (PPE) such as gloves, hardhat, hearing protection and safety glasses (minimal).

15. Chain of custody records and custody seals

4.2 Vial Preparation for VOC Sample Collection

VOC samples will be collected in 40 mL clear glass VOA vials with Teflon-lined septa and screw caps. This sampling requires purge and trap grade methanol for sample preservation. Methanol should be ordered before the start of field work and vials should be prepared in the laboratory prior

to embarking into the field. If vials are prepared in the field it should be in a controlled environment (e.g. with an appropriate balance on a flat surface and in a room free of wind). The following outlines the steps necessary to prepare vials for VOC sample collection of soils:

1. Affix sample label and label each sample container sequentially (e.g. VOC-001, VOC-002, etc.). Label the lid of each sample vial with the same ID as written on the label. Affixing the sample label prior to the sampling event assures the weight of the label will be included in the tare weight of the vial.

2. Check calibration of the balance being used to weigh vials. Record this calibration check in the appropriate calibration logbook.

3. Weigh each empty vial with affixed label and record weights on appropriate forms.

4. Add 10ml of purge and trap grade methanol to each vial, screwing the cap back on each vial after filling it.

5. Re-weigh each vial once 10ml of purge and trap grade methanol has been added to accurately determine the weight and volume of methanol extract. Record these weights on appropriate forms.

6. Sample bottles will be stored at the laboratory in a freezer set between -10°C and -20°C until they are deployed into the field where the will be stored in a cooler with ice or fridge set to 4°C until samples are collected and are shipped to the laboratory.

Manufacturer, lot numbers, and other pertinent information of the methanol used to prepare vials will be recorded on appropriate forms. Recording this information enables Stone to track batches of methanol in the event contamination has partitioned into the methanol from various environmental sources.

4.3 Soil Core Retrieval, Handling, and VOC Sample Collection

Soil cores may be retrieved by several different methods, it is important to select a method that recovers soil cores representative of *in-situ* conditions and minimizes losses of VOCs. Examples of soil coring methods include, but are not limited to Geoprobe® Systems MC5, DT45, DT325/35, or DT22 coring tools, split spoon samplers, and AMS multi-stage soil core samplers. Refer to project specific protocols to determine which soil coring method will be utilized.

The rate of drilling and soil core retrieval shall not exceed the rate at which the sampler can collect samples and describe each core following SEI-6.42*.n Geologic Description of Unconsolidated Deposits*. This will prevent soil cores from being exposed to the atmosphere for extended periods of time prior to sampling, greatly reducing VOC losses.

Upon retrieval of soil cores the following steps shall be followed, working quickly to minimize the loss of VOCs:

1. Cores will be placed on a clean sampling surface, liners or spilt spoons shall be opened to reveal the core, and the core shall be photographed with labels indicating the site ID, borehole ID, depth interval, and recovery.

2. A portable PID will be utilized to screen the soil cores every 6 inches for the possible presence of VOCs. PIDs shall be calibrated daily prior to measurements according to manufactures instructions (SEI-6.19.*n*).

3. VOC sample frequency will follow site specific protocols. In general VOC samples will be collected from strata exhibiting the highest PID responses.

4. Using an appropriate sample device (see section 4.1 of this SOP), collect approximately 10-12 grams (g) of sample as soon as possible after the surface of the soil has been exposed to the atmosphere, generally within a few minutes at most. To the extent practical, samples should be obtained from undisturbed portions of the soil core.

5. Using the sample collection device, add the approximately 10-12g of sample to a pre-prepared sample vial containing purge-and-trap grade methanol.

6. Wipe any soil off the vial threads and immediately seal the vial with the septum and screw cap.

7. If project-specific work plan(s) specify, use a portable balance to weigh the sealed vial containing the sample (after either noting the laboratory-indicated weight or determining the tare weight of the vial in the field) to ensure 10-12g of sample was added. Samples will also be weighed upon receipt at the laboratory to determine sample mass.

8. Using the laboratory supplied label already fixed to the vials, label sample. Do not affix additional labels or custody seals to vials, as this will alter the tare weight.

9. Store samples on ice at 4ºC until delivered to the analytical laboratory.

10. A trip blank comprised of a blank methanol vial should be supplied by the laboratory, and shall be submitted back to the laboratory with each sample delivery group.

11. A separate 10-12g aliquot of soil should be collected for moisture content determination using the same sampling device and methods described above, and placed in an un-preserved container. Label the moisture content sample appropriately.

12. Upon completion of VOC sample collection soil cores shall be described for color, texture, consistence, structure, and moisture, and other parameters as called for in site specific protocols following SEI-6.42.*n*. Collect discrete soil samples for other analytical parameters.

13. Decontaminate reusable sampling equipment prior to reuse according to site specific procedures and/or SEI-5.1.*n Maintenance and Decontamination of Field Equipment.*

4.4 Physical Properties Sample Collection

Soil cores will be retrieved following procedures described in section 4.3 of this SOP. Refer to site specific protocols for sample frequency. However, in general, an effort will be made to obtain samples from different stratigraphic units where variations in stratigraphy as assessed during core logging.

An effort shall be made to collect physical property samples from lesser contaminated sections of coil cores as determined by PID screening.

Each sample will be a cylindrical disc of the same diameter as the soil core retrieved, with a height of approximately 4 to 6 inches. These sections of core will be obtained such that the *in-situ* moisture conditions are retained. Soil samples will be collected for physical property analyses (porosity, bulk density, and *foc*) in the following manner:

1. Select a section of soil core to be sampled
2. Use a hacksaw to cut through plastic core liners on both ends of the desired sample. If split spoons were used carefully roll the soil core onto a clean piece of plastic wrap and remove unwanted soil with a gloved hand or knife.
3. Wrap sample tightly in clean plastic wrap. Tape plastic wrap to secure.
4. If available, place proper size end caps on both ends of sample to secure soil. If not available use additional plastic wrap to secure the core sample.
5. Clearly label each sample with the sample ID, location, depth, and date. Secure label with packing tape and wrap in bubble wrap or other padding.
6. Place samples individually in zip top polyethylene bags.
7. Store samples on ice at 4ºC until delivered to the analytical laboratory.

4.5 Discrete Soil Sample Collection for Other Analytical Parameters

Discrete soil samples may be collected for other analytical parameters including, but not limited to, SVOCs, PAHs, Metals, and PCBs. These samples may be collected from soil cores retrieved from the subsurface using drilling techniques discussed in section 4.3 of this SOP or from surface or shallow soils collected using hand tools, such as a hand auger or AMS multi-stage soil core sampler. These discrete soil samples shall be collected following these steps:

1. Prior to fieldwork contact the laboratory conducting sample analysis and procure appropriate containers for the specific study. Container volume and amount of soil needed may vary by the analyses being conducted.
2. Using a stainless steel spoon or trowel collect the appropriate amount of soil needed for the analyses being performed into the appropriate sample containers provided by the laboratory.
3. Clearly label each sample with the sample ID, location, depth, and date. Secure label with packing tape and wrap in bubble wrap or other padding.
4. Store samples on ice at 4°C until delivered to the analytical laboratory.
5. Decontaminate reusable sampling equipment prior to reuse according to site specific procedures and/or SEI-5.1.*n Maintenance and Decontamination of Field Equipment*.

5.0 RESPONSIBILITIES

1. All personnel will legibly record data and observations (including phone conversations) in accordance with this SOP to enable others to reconstruct project events and provide sufficient evidence of activities conducted.

6.0 DEFINITIONS

1. *Observations & Remarks Form (O&R):* A pre-printed form, which contains mostly blank space for general note taking. The form typically prompts the user for the study or project designation, the SEI project number, the client or sponsor name, the total number of pages (page *n* of n) and requires a signature and date. The form is generally used to capture notes of one person when another, more specific forms is not available.

2. *Raw data* means any worksheets, records, memoranda, notes, or exact copies thereof, that are the result of original observations and activities of a study and are necessary for the reconstruction and evaluation of the report of that study. In the event that exact transcripts of raw data have been prepared (e.g., tapes which have been transcribed verbatim, dated, and verified accurate by signature), the exact copy or exact transcript may be substituted for the original source as raw data. Raw data may include photographs, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments.

3. *VOCs:* Volatile Organic Compounds

4. *SVOCs:* Semi-Volatile Organic Compounds

5. *PAHs*: Polycyclic Aromatic Hydrocarbons

6. *PCBs*: Polychlorinated Biphenyls

7. *PID*: Photoionization Detector

8. *MSDS*: Material Safety Data Sheet

9. *foc*: Fraction of Organic Carbon

7.0 REFERENCES

40 CFR Part 160 Good Laboratory Practice Standards, August, 1989.

8.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

None

9.0 AUTHORIZATION

Written by: _____ Date: _____

Lee Rosberg, Staff Scientist

Approved by: _____ Date: _____

Seth Pitkin, Vice President

10.0 REVISION HISTORY

Not applicable.

Attachment I
Methanol MSDS

STANDARD OPERATING PROCEDURE

SEI-6.46.0

GEOLOGIC DESCRIPTION OF UNCONSOLIDATED DEPOSITS USING THE UNIFIED SOIL CLASSIFICATION SYSTEM

SOP Number: SEI-6.46.0 Date Issued: 08/28/12
Revision Number: 0 Date of Revision: NA

1.0 OBJECTIVE

The objective of this procedure is to provide a standardized geological method of describing the nature of unconsolidated deposits, commonly referred to as soils or sediments, using the Unified Soil Classification System (USCS). A description of unconsolidated geologic deposits is often part of site investigations as a critical element in understanding the properties that govern the movement of water and other fluids, including contaminants. Samples of unconsolidated deposits are typically collected with a split-spoon sampler or other soil coring tools. These samples are then described to aid in interpretations about the geologic and hydrologic characteristics of the site. The characteristics of major importance for this type of description are: soil name, particle size disibution, gradation or plasticity, dry strength, dilatancy, toughness, color, odor, soil texture and structure, moisture content, relative density or consistency, cementation, relative permeability, local geologic name, and group symbol.

The USCS was developed for the US Army Corps of Engineers and Bureau of Reclamation for classifying soils for engineering purposes based on laboratory determination of particle size, liquid limit and plasticity index. It was first used to judge a soil's suitability as a subgrade for roads and airfields, but it is used today for most engineering applications of soil. It differentiates soils into three major divisions: coarse-grained, fine-grained and highly organic soils as shown in Attachment 1. Fine-grained soils are classified as those that will pass through a No. 200 U.S. standard sieve (0.075 mm). Organic material is a common component of soil but it has no size range. Each type of soil is given a two-letter designation based primarily on its particle-size distribution (texture), Atterberg limits, and organic matter content. Attachments 1 and 2 describe the USCS (reference).

2.0 POLICIES

1. The Staff Scientist will follow the USCS method of describing the nature of the unconsolidated deposits and will document their fieldwork in a field notebook for all site investigations.
2. Staff Scientists will be properly trained in site investigations prior to conducting a site investigation as documented in their training record on file at Stone Environmental, Inc.

3.0 SAFETY ISSUES

Staff following the methodology outlined in this SOP will read the site Health and Safety plan for the individual project, as well as appropriate Material Safety Data Sheets (MSDS).

4.0 PROCEDURES

4.1 Equipment

1. A notebook computer, field notebook, logging forms or other acceptable recording medium.
2. Indelible pens
3. A measuring rule in feet and tenths and hundreds of feet (not inches)
4. Measuring devices, such as engineers scale, electronic balances, electronic water level indicator, interface probe, etc.
5. A rule in mm for measuring particle sizes
6. A grain size key
7. Hand lens
8. Diluted Hydrochloric acid (HCl)
9. Knife or other appropriate tool for handling soil
10. Clean (new) poly/plastic sheeting or other material on which collected samples may be examined/preserved.
11. Appropriate personal protective equipment.
12. Sample containers, forms, labels, markers, preservatives and shipping materials.

4.2 Soil Description Procedures

4.2.1 Separating Significant Layers

Each distinct sediment layer in the split spoon or other type of sample should be described separately. Layers may be differentiated based on any of the characteristics listed in section 1.0. It may be most appropriate to describe layers less than 0.5 inches thick as laminae. In a situation where several laminae of similar properties are located in a sample, it may be best to describe them as a group. For example, 8 gray silty clay laminae between 5.5 and 6.5 feet, each about 1/4 inch thick. The following sections discuss each of the soil characteristics to be described.

4.2.1.1 Angularity

Describe the angularity of the sand (coarse sizes only), gravel, cobbles, and boulders, as angular, subangular, subrounded, or rounded in accordance with the criteria in Table 3 and Fig. 1, below. A range of angularity may be stated, such as: subrounded to rounded.

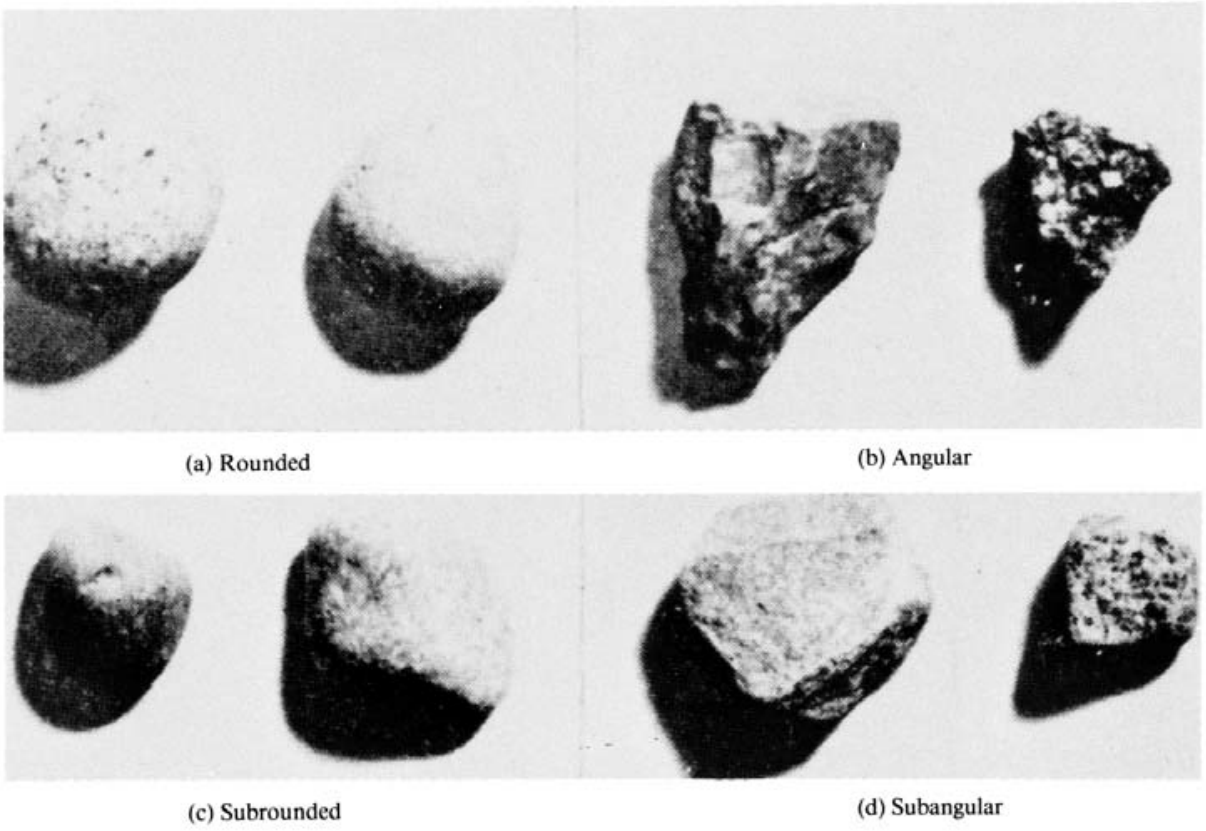
Table 3

Criteria for Describing Angularity of Coarse-Grained Particles (See Fig.1)

Description	Criteria
Angular	Particles have sharp edges and relatively plane sides with unpolished surfaces
Subangular	Particles are similar to angular description but have rounded edges
Subrounded	Particles have nearly plane sides but have well-rounded corners and edges
Rounded	Particles have smoothly curved sides and no edges

Figure 1

Typical Angularity of Bulky Grains



4.2.1.2 Shape

Describe the shape of the gravel, cobbles, and boulders as flat, elongated, or flat and elongated if they meet the criteria in Table 4 and Fig. 2, below. Otherwise, do not mention the shape. Indicate the fraction of the particles that have the shape, such as: one-third of the gravel particles are flat.

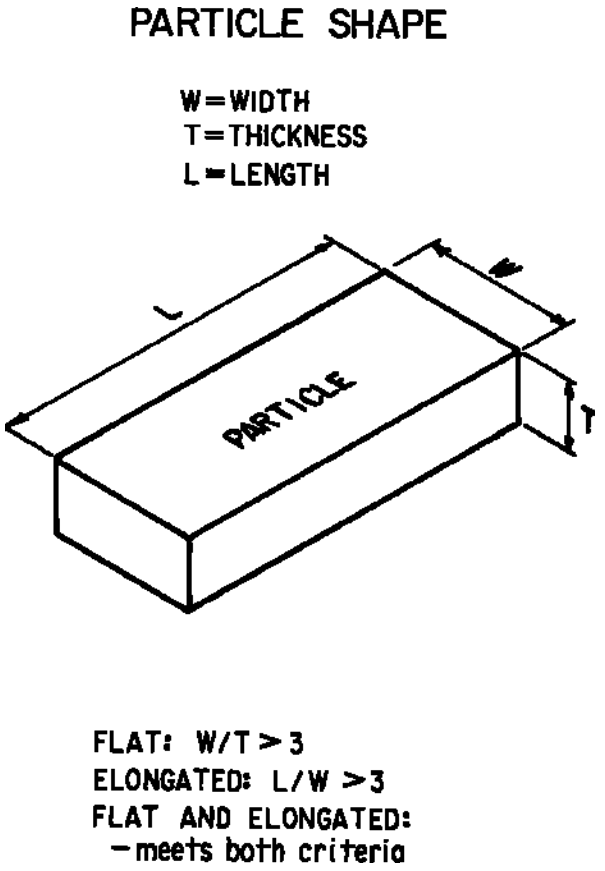
Table 4

Criteria for Describing Particle Shape (see Fig. 2)

The particle shape shall be described as follows where length, width, and thickness refer to the greatest, intermediate, and least dimensions of a particle, respectively.	
Flat	**Particles with width/thickness > 3**
Elongated	**Particles with length/width > 3**
Flat and Elongated	**Particles meet criteria for both flat and elongated**

Figure 2

Criteria for Particle Shape



4.2.1.3 Color

Describe the color. Color is an important property in identifying organic soils, and within a given locality it may also be useful in identifying materials of similar geologic origin. If the sample contains layers or patches of varying colors, this shall be noted and all representative colors shall be described. The color shall be described for moist samples. If the color represents a dry condition, this shall be stated on the soil boring log.

4.2.1.4 Odor

Describe the odor if organic or unusual. Soils containing a significant amount of organic material usually have a distinctive odor of decaying vegetation. This is especially apparent in fresh samples, but if the samples are dried, the odor may often be revived by heating a moistened sample. If the odor is unusual (petroleum product, chemical, and the like), it shall be described.

4.2.1.5 Moisture Condition

Describe the moisture conditionas dry, moist, or wet, in accordance with the criteria in Table 3, below.

Table 3

Criteria for Describing Moisture Condition

Description	Criteria
Dry	Absence of moisture, dusty, dry to the touch
Moist	Damp but no visible water
Wet	Visible free water, usually soil is below water table

4.2.1.6 HCL Reaction

Describe the reaction with HCl as none, weak, or strong, in accordance with the critera in Table 4, below. Since calcium carbonate is a common cementing agent, a report of its presence on the basis of the reaction with dilute hydrochloric acid is important.

Table 4

Criteria for Describing the Reaction with HCl

Description	Criteria
None	No visible reaction
Weak	Some reaction, with bubbles forming slowly
Strong	Violent reaction, with bubbles forming immediately

4.2.1.7 Consistency

For intact fine-grained soil, describe the consistency as very soft, soft, firm, hard, or very hard, in accordance with the criteria in Table 5, below. This observation is inappropriate for soils with significant amounts of gravel.

Table 5

Criteria for Describing Consistency

Description	Criteria
Very Soft	Thumb will penetrate soil more than 1 in. (25 mm)
Soft	Thumb will penetrate soil about 1 in. (25 mm)
Firm	Thumb will indent soil about 1⁄4 in. (6 mm)
Hard	Thumb will not indent soil but readily indented with thumbnail
Very Hard	Thumbnail will not indent soil

4.2.1.8 Cementation

Describe the cementation of intact coarse-grained soils as weak, moderate, or strong, in accordance with the criteria in Table 6, below.

Table 6

Criteria for Describing Cementation

Description	Criteria
Weak	Crumbles or breaks with handling or little finger pressure
Moderate	Crumbles or breaks with considerable finger pressure
Strong	Will not crumble or break with finger pressure

4.2.1.9 Structure

Describe the structure of intact soils in accordance with the criteria in Table 7, below.

Table 7

Criteria for Describing Structure

Description	Criteria
Stratified	Alternating layers of varying material or color with layers at least 6 mm thick; note thickness
Laminated	Alternating layers of varying material or color with the layers less than 6 mm thick; note thickness
Fissured	Breaks along definite planes of fracture with little resistance to fracturing
Slickensided	Fracture planes appear polished or glossy, sometimes striated
Blocky	Cohesive soil that can be broken down into small angular lumps which resist further breakdown
Lensed	Inclusion of small pockets of different soils, such as small lenses of sand scattered through a mass of clay; note thickness
Homogenous	Same color and appearance throughout

4.2.1.10 Range of Particle Sizes

For gravel and sand components, describe the range of particle sizes within each component as defined in 3.1.2 and 3.1.6. For example, about 20 % fine to coarse gravel, about 40 % fine to coarse sand.

The consistence of the sediment will be described whenever it remains evident in the split spoon sample. The sample may be compacted excessively or otherwise disturbed during sampling and the natural consistence may not be evident during examination. If the

consistence can be characterized, it will be described according to the terms and definitions listed in Attachment 4.

The key distinction for this parameter is the change from friable to firm. When handling a sample that is friable, it will take only slight pressure between the thumb and fingers to cause a clod or ped of the sediment to break apart. A sample that is firm will resist crushing to the point where it finally seems to explode when it is subjected to adequate pressure. A friable clod or ped will not explode or pop, it will simply fall apart when pressure is applied. The other descriptive classes, as show in Attachment 4, will be applied to add more information as to the degree of soil stability, or lack thereof.

4.2.1.11 Structure

Observations of structure will include a characterization of the orientation and continuity of contacts (e.g., sharp or gradational), angularity of grains (see Attachment 3), presence of fractures, cracks, partings or macropores and a qualitative description of the degree of sorting (poorly sorted means many different grain sizes, well sorted means uniform grain size). Additional description of such as features as crossbeds, grading, ripples and varves will aid in identifying the depositional environment.

4.2.1.12 Moisture

The soil moisture will be described on the basis of visual and manual field observations. The moisture class will be based on the percent water in the soil as follows:

Moisture Class	Moisture Content (%)	Field Description
Dry	0	No dampness is evident in sample.
Humid	1-25	Slight damp mark seen on hand when sample is squeezed.
Moist	26-75	Grading from humid to wet.
Wet	76-99	Water is released when sample is squeezed.
Saturated	100	Water drips freely from sample.

4.2.1.13 Concretions

When iron or manganese concretions are present in a soil sample, they will be noted as to their abundance and size. The use of dilute hydrochloric acid is helpful in identifying the presence of carbonate minerals, though its use is not required.

4.2.1.14 Other Parameters

The protocols or work plan for a given study may require the determination of other parameters, e.g., screening for the presence of volatile organic compounds with a photoionization detector at a hazardous waste investigation. In such cases, the protocol, work plan, additional SOPs, or other authority or regulation will describe the additional or alternate method of sediment evaluation.

4.3 Data Recording

The information required by this SOP will be recorded in a field book during the sampling process. Required information includes:

Project name

SEI study number

Site name

Well/Profile ID number

Name of geoscientist

Driller's name

Date of exploration

Attachment 5 presents a format for recording field data as well as examples of the descriptors to be used. All of the data fields must be addressed for each discrete layer being described. All measurements should be in feet and tenths of feet (not inches). The lengths of the segments of the particular sample being described must add up to the linear footage of recovery noted. In the interest of standardization, if less than complete recovery is achieved it should be assumed that the missing portion of the sample interval is at the bottom of the sample.

5.0 RESPONSIBILITIES

5.1 Principals/Group Officers/Group Leaders/Personnel

Any staff conducting these procedures are responsible for signing, dating, and managing all field observation forms.

6.0 DEFINITIONS

1. *Concretions* A nodular or irregular concentration of certain authigenic constituents of sedimentary rocks and tuffs; developed by the localized deposition of material from solution, generally about a nucleus. Harder than enclosing rock.
2. *Lamina (ae)* Unit layer or sheet of sediment in which the stratification planes are one centimeter or less apart. Laminae need not be parallel to bedding. When they are at an angle to the bedding planes the term "cross-lamination" may be used.

7.0 REFERENCES

Davis, Richard A. Jr., 1992, Depositional Systems: An Introduction to Sedimentology and Stratigraphy, 2nd Edition, Prentice Hall, New Jersey, 604 p.

Hunter/BCA. Standard Operating Procedure No. 6006, Identification of Soils

American Geological Institute, 1976, Dictionary of Geological Terms, Revised Edition, Anchor Press/Doubleday, Garden City, New York, 472 p.

Laboratory Manual for Hand Specimen Petrology, Department of Geology, University of Texas [Undated].

8.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

1. Table 1: Unified Soil Classification System Description #1
2. Table 2: Unified Soil Classification System Description #2
3. Table 3: Criteria for Describing Angularity of Coarse-Grained Particles
4. Table 4: Criteria for Describing Particle Shape
5. Figure 1:

9.0 AUTHORIZATION

Revised by: _____ Date:_____

Lee Rosberg, Project Scientist

Approved by:_____ Date:_____

Christopher T. Stone, President

10.0 REVISION HISTORY

Revision 1

1. Renumbered from Chapter SEI-10.11.1

Revision 2

1. Changed title from geologic description of unconsolidated deposits to geologic description of unconsolidated deposits using the Wentworth grain size scale
2. Added second paragraph of section 1.0
3. Updated Attachment 5 with current soil boring log

Several minor grammatical edits

Table 1

Unified Soil Classification System Description #1

Unified Soil Classification System; from American Society for Testing and Materials, 1985				
Major Divisions			Group Sym.	Group Name
Coarse Grained Soils–More Than 50% Retained On No.200 Sieve	Gravel–More Than 50% of Coarse Fraction Retained On No.4 Sieve	Clean Gravel	GW	Well-Graded Gravel, Fine to Coarse Gravel
			GP	Poorly-Graded Gravel
		Gravel With Fines	GM	Silty Gravel
			GC	Clayey Gravel
	Sand–More Than 50% of Coarse Fraction Passes No.4 Sieve	Clean Sand	SW	Well-Graded Sand, Fine to Coarse Sand
			SP	Poorly-Graded Sand
		Sand With Fines	SM	Silty Sand
			SC	Clayey Sand
Fine Grained Soils–More Than 50% Passes No. 200 Sieve	Silt And Clay Liquid Limit Less Than 50	Inorganic	ML	Silt
			CL	Clay
		Organic	OL	Organic Silt, Organic Clay
	Silt And Clay Liquid Limit 50 Or More	Inorganic	MH	Silt of High Plasticity, Elastic Silt
			CH	Clay of High Plasticity, Fat Clay
		Organic	OH	Organic Clay, Organic Silt
Highly Organic Soils			Pt	Peat

Table 2

Unified Soil Classification System Description #1

Unified Soil Classification System (USCS)			
	Millimeters	Inches	Sieve Size
Boulders	> 300	> 11.8	-
Cobbles	75 - 300	2.9 - 11.8	-
Gravel:			
Coarse	19 - 75	0.75 - 2.9	-
Fine	4.8 - 19	0.19 - 0.75	3/4" - No. 4
Sand:			
Coarse	2.0 - 4.8	0.08 - 0.02	No. 4 - No. 10
Medium	0.43 - 2.0	0.02 - 0.08	No. 10 - No. 40
Fine	0.08 - 0.43	0.003 - 0.02	No. 40 - No. 200
Fines:			
Silts	< 0.08	< 0.003	< No. 200
Clays	< 0.08	< 0.003	< No. 200

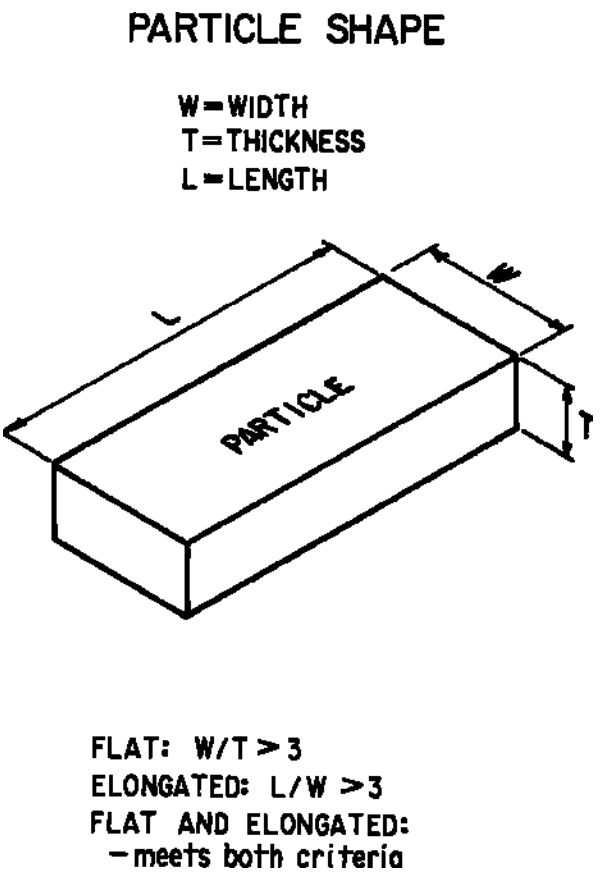
Table 4

Criteria for Describing Particle Shape (see Fig. 2)

The particle shape shall be described as follows where length, width, and thickness refer to the greatest, intermediate, and least dimensions of a particle, respectively.	
Flat	Particles with width/thickness > 3
Elongated	Particles with length/width > 3
Flat and Elongated	Particles meet criteria for both flat and elongated

Figure 2

Criteria for Particle Shape

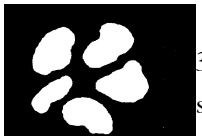


ATTACHMENT 3

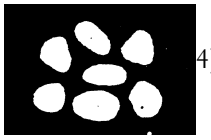
ANGULARITY OF ROCK FRAGMENTS

1) Angular: having sharp corners and edges, and therefore showing little or no effects of abrasion or wear.

2) Subangular: having edges and corners slightly rounded, so that wear is evident.



3) Subround: having most of the corners and edges worn down to smooth curves, and thus showing extensive abrasion.



4) Round: having all edges and corners smoothed off to gentle curves by prolonged wear.

ATTACHMENT 4
CONSISTENCE CLASSES

	Class	Description
Dry Soil:		
	dl —	loose- usually associated with sands
	ds —	soft- fragile
	dsh —	slightly hard- breaks easily
	dh —	hard- barely breakable with fingers
	deh —	extremely hard- can't break with fingers
Moist Soil:		
	ml —	noncoherent
	mvfr —	very friable- very easily broken apart
	mfr —	friable- easily broken apart
	mfi —	firm- resist crushing, "explode" when finally crushed
	mvfi —	very firm- barely breakable with fingers
Wet Soil:		
	wso —	nonsticky
	wss —	slightly sticky- soil sticks to one finger when squeezed and separated
	ws —	sticky- soil sticks to both thumb and finger, stretches slightly
	wvs —	very sticky- soil sticks strongly, resists separation of thumb and finger
	wsp —	slightly plastic- forms wire when rolled between hands, but breaks easily
	wp —	plastic- forms wire when rolled between hands, doesn't break easily
	wvp —	very plastic- forms a very durable wire when rolled between hands

ATTACHMENT 5
SOIL BORING LOG

STANDARD OPERATING PROCEDURE

SEI-6.47.0

NON-AQUEOUS PHASE LIQUID (NAPL) SAMPLING

SOP Number: SEI-6.47.0
Revision Number: NA

Date Issued: 10/16/12
Date of Revision: NA

1.0 OBJECTIVE

This standard operating procedure outlines the sampling methods to be used for collection of NAPLs including light, non-aqueous-phase liquids (LNAPLs) and dense, non-aqueous-phase liquids (DNAPLs) from monitoring wells and other water columns. LNAPLs are generally non-aqueous immiscible organic liquids that have densities less than water. These chemicals, which include petroleum products, tend to float on the groundwater surface and commonly occupy the capillary fringe zone above the water table. DNAPLs include immiscible organic liquids, such as chlorinated solvents, PCBs, and coal tar, which have densities greater than water. DNAPLs tend to migrate downward through the saturated zone until they encounter lower permeability beds within the aquifer. Measurement of the thickness of the NAPL must be performed prior to sampling.

This procedure is also applicable to the collection of dense, aqueous phase liquids (DAPLs), which generally comprise inorganic brines, although certain features, such as a sharp water/DAPL boundary, are not relevant.

2.0 POLICIES

1. Personnel will legibly record data any observations in the field to enable others to reconstruct project events and provide sufficient evidence of activities conducted.
2. Field personnel will use a field logbook, observation and remark form (O&R) or other designated field form to record activities, measurements, and observations made in the field.

3.0 SAFETY ISSUES

1. Whenever visiting a hazardous or potentially hazardous site, it is important to read, follow and sign the site health and safety protocol (HASP). Sampling and handling of NAPLs requires special precautions and selection of appropriate personal protective equipment (PPE).
2. Field visits may involve accessing remote areas. Health and safety concerns regarding these field visits are minimal however; slip, trip and fall hazards, and poisonous plant and dangerous animal, as well as getting vehicles stuck in remote areas do present considerable health and safety issues. To help ensure field staff's health and safety with remote areas, all field staff is to have daily communication with the project manager or another appropriate Stone Environmental, Inc. (Stone) employee.

4.0 PROCEDURES

4.1 Equipment and Materials

1. Oil/water interface probe.
2. Clean, inert string, not to be used in another well.
3. PID.
4. Top filling bailer or single check valve bailer-LNAPL.
5. Single or dual check valve bailer-DNAPL.
6. Peristaltic pump and polyethylene tubing-DNAPL.
7. Bacon Bomb Sampler-DNAPL (Optional).

   Note: One dedicated bailer for each well is preferred. If a bailer is to be shared between wells, make sure it can be dismantled and thoroughly decontaminated in the field. Disposable bailers commonly have seams that can collect contaminants and are impossible to clean; these bailers should not be used in more than one well. Bailers must be constructed of materials that will not react with compounds to be analyzed.

8. Protective gloves appropriate for site safety concerns and sample integrity.
9. Sample containers for water quality measurements.
10. Calibrated purge buckets.
11. Field notebook with protocol, applicable SOPs, study specific procedures, sampling forms, Chain of custody, and or other appropriate medium for recording field data.
12. Sample containers and labels.
13. Well keys.
14. Electrical tape.
15. Clear packing tape.
16. Distilled water.
17. Alconox.
18. Spray bottles for decontamination.

4.2 Procedure

The Project Manager or field personnel will contact the receiving laboratory that high concentration samples will be shipped to the facility and that separate storage from regular samples will be required. In addition, the chain-of custody accompanying these samples must document that these samples are high concentration samples and shipping containment must be separate from other samples from the site. When appropriate or necessary, all fittings, equipment, and receptacles used in the sampling process must be decontaminated in accordance with standard operating procedures for the decontamination of field equipment (see SOP 5.1.n). If volatile organic analysis is required a trip blank will accompany all samples in accordance with the site quality assurance plan or work plan.

4.2.1 LNAPL Sample Collection

Measurement of the thickness of the floating layer may be determined by using an oil/water interface probe, tape with indicator paste (Kolor Kut Gasoline Gauging Paste), or clear bailer. The most widely tested and used oil/water detection probe employs a combination of optical and electrical conductivity measurements. This combination allows detection of air- liquid, floating product-water, and water-sinking product interfaces. The thickness of each product layer is obtained by subtracting the difference between subsequent interfaces as measured on the calibrated lowering tape. Indicator paste absorbs petroleum products and indicates this by a change in color. The tape is left in place for several seconds, and then retrieved. The distance between the starting and end points of the color change indicate the product thickness. If a clear bailer is used to measure product thickness, it must be long enough to cover the entire thickness of the floating product.

Prior to the purging of groundwater from the well, a sample of the floating layer may be obtained using bottom filling bailer or top filling bailer.

If more than one distinct LNAPL layer is present in a well, each layer should be sampled. Samples should be analyzed for chemical composition and physical parameters (e.g., specific gravity, viscosity, water solubility, etc.).

4.2.1.1 Bottom-Filling Bailers

Single check valve, bottom-filling bailers are the most commonly utilized bailer. A single ball check is seated at the bottom of the bailer. The act of lowering the bailer through the water column opens the check valve and liquid fills the bailer from the bottom. Upon retrieval, the weight of the liquid inside the bailer closes the check valve as the bailer exits the liquid column. The liquids in the bailer are retained from the greatest depth to which the bailer was lowered. The following procedure shall be used when utilizing a bottom filling bailer:

1. Put on clean sampling gloves.

2. Obtain a clean bailer. Do not allow the bailer to come into contact with surface soils on the site.
3. Attach the inert string to the bailer. Make sure the knot is secure and that there is a sufficient length of string to reach the bottom of the well.
4. Slowly lower the bottom of the bailer to just above the lower depth of measured LNAPL.
5. Raise the bailer out of the well, coiling the string so as not to let it come into contact with the ground.
6. Allow entrained water to separate from LNAPL and decant water from bailer.
7. Decant LNAPL into appropriate containers. Label containers with appropriate sample information.
8. Repeat steps 4 through 7 as necessary to obtain sufficient sample volume.
9. Place filled sample containers on ice immediately.
10. Record the information in the field logbook and complete all chain-of-custody records (notifying the receiver that these samples are high concentration samples and must be stored separately) and field sheets.
11. Decontaminate the sampler, if necessary.

4.2.1.2 Top Filling Bailers

Collecting LNAPLs with bottom-filling bailers is difficult when the thickness of the LNAPL layer is less than the submergence required to lift the ball check off the seat of the inlet valve to allow liquid to enter the bailer. In these cases, a bottom-filling bailer may be unable to effectively collect samples of thin LNAPL layers. In contrast, a top-filling bailer collects from the surface of a liquid and fills by skimming the surface. When using a top-filling bailer to collect LNAPL follow these steps:

1. Put on clean sampling gloves.
2. Obtain a clean bailer. Do not allow the bailer to come into contact with surface soils on the site.
3. Attach the inert string to the bailer. Make sure the knot is secure and that there is a sufficient length of string to reach the bottom of the well.
4. Slowly lower the bailer such that the top of the bailer is just below the top of measured LNAPL, taking care to not allow the bailer to fill beyond the estimated volume of LNAPL in the layer, as this will result in the LNAPL initially skimmed from the surface to be displaced by water.
5. Raise the bailer out of the well, coiling the string so as not to let it come into contact with the ground.
6. Allow entrained water to separate from LNAPL.

7. Decant LNAPL into appropriate containers. Label containers with appropriate sample information.
8. If additional sample volume is required, allow time for LNAPL to recharge in the well, and repeat Steps 4 through 7 as necessary.
9. Place filled sample containers on ice immediately.
10. Record the information in the field logbook and complete all chain-of-custody records (notifying the receiver that these samples are high concentration samples and must be stored separately) and field sheets.
11. Decontaminate the sampler, if necessary.

4.2.1.3 Peristaltic Pumps

Peristaltic pumps may be used for LNAPL sample collection where the depth to liquids in the well or water column do not exceed the suction limit (approximately 25 feet below ground surface). To determine tubing intake depth measure LNAPL thickness using an oil/water interface. For collection of LNAPL samples using a peristaltic pump follow these steps:

1. Lower clean poly tubing to bottom of the well.
2. Attach tubing sample tubing to clean flexible silicon pump tubing.
3. Attach a short piece of clean polyethylene tubing to discharge side of pump.
4. Ensure pump speed is set on slowest speed and turn pump on.
5. Discharge LNAPL into appropriate sample containers. When desired amount of sample is collected turn pump off, cap and label sample containers.

4.2.2 DNAPL/DAPL Sample Collection

Measurement of DNAPL thickness may be accomplished using an interface probe as described above for LNAPLs. Due to their greater solubility, measurement of DAPL thickness is more problematic, as no defined boundary exists.

A DNAPL sample may be obtained by utilizing a Bacon Bomb sampler, single check valve bailer, dual check valve bailer, or tubing attached to a peristaltic pump. Samples should be analyzed for chemical composition and physical parameters (e.g., specific gravity, viscosity, water solubility, etc.).

4.2.2.1 Bacon Bomb Sampler:

The Bacon Bomb Sampler is a widely used, commercially available sampler, designed for sampling petroleum products and viscous liquids. It is useful in collecting samples at various vertical locations in a liquid column. The Bacon Sampler remains unopened until it reaches the desired sampling depth. The Bacon Sampler is difficult to decontaminate, and it is difficult to transfer the sample into

the sample bottles. The possibility of aerating the sample exists if the sampler does not completely fill with water and air is entrapped in the sampler during retrieval.

The Bacon Bomb Sampler is constructed of brass or stainless steel and is available in two sizes: 1.5 in. or 3.5 in. diameters. Samplers range in volume from 250 mL to 1 liter. The Bacon Bomb Sampler is equipped with a spring-loaded trigger. When activated, the trigger allows liquid to enter the collection chamber. When the trigger is released, liquid is prevented from flowing into or out of the collection chamber. The following procedure shall be followed when using a Bacon Bomb Sampler to collect DNAPL samples:

1. Carefully lower the Bacon Bomb Sampler with an inert string to the bottom of the well or water column, allowing the line for the trigger to remain slack at all times.
2. When the bottom of the well is reached, pull the trigger line until taut. Wait a few seconds to allow the sampler to fill with liquid.
3. Release the trigger line and retrieve the sampler.
4. Transfer the sample to the sample bottles by pulling on the trigger, being careful to exclude water that may have been entrained in the sampler. Allow the sample to flow down the side of the sample bottle with minimal disturbance.
5. If additional sample volume is required, allow time for DNAPL to recharge in the well, and repeat Steps 1 through 4 as necessary.
6. Secure the sample bottle cap tightly.
7. Label the sample bottle with an appropriate label. Be sure to complete the label with all necessary information.
8. Place filled sample containers on ice immediately.
9. Record the information in the field logbook and complete all chain-of-custody records (notifying the receiver that these samples are high concentration samples and must be stored separately) and field sheets.
10. Decontaminate the sampler.

4.2.2.2 Bailers

Single Check Valve Bailers:

Single check valve bailers, as described in section 4.2.1 of this SOP, can be utilized to sample DNAPL from monitoring wells or other water column. Follow steps outlined below for DNAPL sample collection using a single check valve bailer:

1. Put on clean sampling gloves.
2. Obtain a clean bailer. Do not allow the bailer to come into contact with surface soils on the site.

3. Attach the inert string to the bailer. Make sure the knot is secure and that there is a sufficient length of string to reach the bottom of the well.
4. Lower the bailer to the bottom of the well on the nylon string, and allow the bailer to fill with DNAPL, which will displace water that has previously filled the bailer.
5. Raise the bailer out of the well, coiling the string so as not to let it come into contact with the ground.
6. Allow entrained water to separate from DNAPL.
7. Decant DNAPL into appropriate containers.
8. If additional sample volume is required, allow time for DNAPL to recharge in the well, and repeat Steps 2 through 7 as necessary.

Dual Check Valve Bailers:

Dual Check valve bailers are similar to single check valve bailers, except an additional check valve is located at the top of the bailer. In single check valve bailers, there is potential for the contents of the bailer to mix with the surrounding water column during deployment or retrieval, depending on the design of the bailer top. A dual check valve bailer is designed to prevent mixing of the sample with the water column upon retrieval. Like the single check valve bailer, water passes through the bailer as it is lowered; however upon retrieval both check valves seat, ideally retaining a depth-discrete aliquot of DNAPL inside the bailer. Follow steps outlined above for DNAPL sample collection using a dual check valve bailer.

4.2.2.3 Peristaltic Pumps

Peristaltic pumps may be used for DNAPL sample collection where the depth to liquids in the well or water column do not exceed the suction limit (approximately 25 feet below ground surface). For collection of DNAPL samples using a peristaltic pump follow these steps:

6. Lower clean poly tubing to bottom of the well.
7. Attach tubing sample tubing to clean flexible silicon pump tubing.
8. Attach a short piece of clean polyethylene tubing to discharge side of pump.
9. Ensure pump speed is set on slowest speed and turn pump on.
10. Discharge DNAPL into appropriate sample containers. When desired amount of sample is collected turn pump off, cap and label sample containers.

4.3 Documentation

The sample container should be labeled, at a minimum, with the following information. (Generally, it is easier to attach the label or write on the container before collecting the sample. Condensation on the containers forms after sample collection, and reduces the likelihood of tape or ink sticking):

1. Study number(s) or project name.
2. Sample identification (including the type of sample - groundwater).
3. Date of sample collection.

Secure the label with a wrap of clear tape if applicable. Follow protocol or Quality Assurance Project Plan requirements for further label needs.

If appropriate to the study, the cap of the bottle can be secured with tape to prevent accidental loss of sample during storage and shipment.

Place the samples out of direct sunlight as soon as possible or into the appropriate storage devices such as a cooler or boxed container.

A record of the sample collection must be made in the field at the time the sample is collected. The record should be made in a field notebook, a sample collection form, or other medium acceptable according to the study protocol or work plan. The information to be recorded must include the following:

1. Date and time of sample collection.
2. Name(s) of the personnel performing the sampling.
3. Sample location, well identification, and / or sample identification.
4. Project/study designation.
5. Depth to liquids prior to sampling (equilibrium level).
6. Method of sample collection.
7. Note on the chain of custody notifying the receiver that these samples are high concentration samples and must be stored separately.

5.0 RESPONSIBILITIES

1. All personnel will legibly record data and observations (including phone conversations) to enable others to reconstruct project events and provide sufficient evidence of activities conducted.

6.0 DEFINITIONS

1. *Observations & Remarks Form (O&R):* A pre-printed form, which contains mostly blank space for general note taking. The form typically prompts the user for the study or project designation, the Stone project number, the client or sponsor name, the total number of pages (page *n* of n) and requires a signature and date. The form is generally used to capture notes of one person when another, more specific forms is not available.
2. *Raw data* any laboratory worksheets, records, memoranda, notes, or exact copies thereof, that are the result of original observations and activities of a study and are necessary for the reconstruction and evaluation of the report of that study. In the event that exact transcripts of raw data have been prepared (e.g., tapes which have been transcribed verbatim, dated, and verified accurate by signature), the exact copy or exact transcript may be

substituted for the original source as raw data. Raw data may include photographs, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments.

3. *LNAPL:* Light, Non-Aqueous Phase Liquid
4. *DNAPL*: Dense, Non-Aqueous Phase Liquid
5. *DAPL*: Dense, Aqueous Phase Liquid

7.0 REFERENCES

40 CFR Part 160 Good Laboratory Practice Standards, August, 1989.

8.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

Attachment 1: Example Monitoring Well Sampling Form

9.0 AUTHORIZATION

Written by: Lee Rosberg Date: 10/16/12

Lee Rosberg, Project Scientist

Approved by: Christopher T. Stone Date: 10/16/12

Christopher T. Stone, President

10.0 REVISION HISTORY

Not Applicable

Attachment I

Example Monitoring Well Sampling Form

STONE ENVIRONMENTAL INC

MONITORING WELL SAMPLING FORM

Project Name:	
SEI Project Number:	
Client:	
Project Manager:	

WELL ID			
Sample Date			
SOP/SSP #'s Followed			SEI Equipment ID
Sampling Method	Bladder Pump ___ Peri Pump ___ Whale Pump___ Bailer ___ Other _____	Wtr Level Indicator	
Sampling Personnel		Water Quality Sonde SN	
Weather			

Calculate Purge Volumes

Time of water level measurement (military): Depth of Pump/Intake: _____ feet

Total Well Depth (btoc)	Depth to Water (btoc)	Height of Water Column		One Well Volume
_____ feet	_____ feet	= _____ feet	X 0.155 liters/feet (1-inch well) X 0.617 liters/feet (2-inch well)	= _____ liters

3 X One Well Volume	liters
5 X One Well Volume	liters

Water Level (ft btoc)	Cumulative Vol. Purged (mL)	Time (Military)	Flow Rate (mL/min)	Temp (°C)	ORP (mV) (+/- 10 mV)	pH (su) (+/- 0.1 su)	DO (mg/L) (+/- 10%)	Conductivity (µS) (+/- 3%)	Turbidity (NTU)

Total Vol. Removed: _____ Liters (√) ___Meters Calibrated (√) ___Min. 3 Well Vol. Purged (√) ___Parameters Stable for 3 consecutive measurements

Sample Number	Time Collected (Military)	Approx. Vol. (mL)	Sampled By (Initials)	Comments:

Sampling Personnel Signature _____ Date _____

STANDARD OPERATING PROCEDURE

SEI-6.42.2

GEOLOGIC DESCRIPTION OF UNCONSOLIDATED DEPOSITS USING THE WENTWORTH GRAIN SIZE SCALE

SOP Number: SEI-6.42.2
Revision Number: 2

Date Issued: 01/18/02
Date of Revision: 10/16/12

1.0 OBJECTIVE

The objective of this procedure is to provide a standardized geological method of describing the nature of unconsolidated deposits, commonly referred to as soils or sediments, using the Wentworth grain size scale. A description of unconsolidated geologic deposits is often part of site investigations as a critical element in understanding the properties that govern the movement of water and other fluids, including contaminants. Samples of unconsolidated deposits are typically collected with a split-spoon sampler or other soil coring tools. These samples are then described to aid in interpretations about the geologic and hydrologic characteristics of the site. The characteristics of major importance for this type of description are: color, texture (e.g., grain size, sphericity, and roundness), consistence, structure, moisture content and concretions.

The Wentworth scale was developed in 1922 and is based on the work of Udden. It is the generally accepted standard used by geologists and sedimentologists in North America (Pettijohn, 1975). It is a logarithmic scale in that each grade limit is twice as large as the next smaller grade limit (Folk, 1974). It is used to describe the texture of sedimentary rocks (e.g., sandstone) as well as unconsolidated sediments.

2.0 POLICIES

1. The Staff Scientist will follow the standardized geological method of describing the nature of the unconsolidated deposits (unless otherwise noted) and will document their fieldwork in a field notebook for all site investigations.
2. Staff Scientists will be properly trained in site investigations prior to conducting a site investigation as documented in their training record on file at Stone Environmental, Inc.

3.0 SAFETY ISSUES

Staff following the methodology outlined in this SOP will read the site Health and Safety plan for the individual project, as well as appropriate Material Safety Data Sheets (MSDS).

4.0 PROCEDURES

4.1 Equipment

1. A notebook computer, field notebook, logging forms, observations and remarks form or other acceptable recording medium.
2. Indelible pens
3. A measuring rule in feet and tenths and hundreds of feet (not inches)
4. Measuring devices, such as engineers scale, electronic balances, electronic water level indicator, interface probe, etc.
5. A rule in mm for measuring particle sizes
6. A grain size key
7. Knife or other appropriate tool for handling soil
8. Clean (new) poly/plastic sheeting or other material on which collected samples may be examined/preserved.
9. Appropriate personal protective equipment.
10. Sample containers, forms, labels, markers, preservatives and shipping materials.

4.2 Geologic Description Methodology

4.2.1 Separating Significant Layers

Each distinct sediment layer in the split spoon or other type of sample should be described separately. Layers may be differentiated based on any of the characteristics listed above. It may be most appropriate to describe layers less than 0.5 inches thick as laminae. In a situation where several laminae of similar properties are located in a sample, it may be best to describe them as a group. For example, 8 gray silty clay laminae between 5.5 and 6.5 feet, each about 1/4 inch thick. The following sections discuss each of the soil characteristics to be described.

4.2.1.1 Color

For most studies, a very general assessment of color based on simple observation is sufficient, but in some cases a study protocol may require that color be measured by comparing the soil to Munsell color charts. The Munsell system designates colors according to three variables: Hue, Value, and Chroma. The hue of a color indicates its relation to red, yellow, green, blue, or purple. The value indicates the lightness of the color, and the chroma indicates the strength of the color.

Stone Environmental uses eight pages of Munsell charts, displaying 251 separate color chips. Each page contains color chips of the same hue, ranging from reddish hues to yellow-green hues and including a one page "gley chart, which has colors in the green-blue-grey range.

Each hue is designated by a number followed by one or two letters. The eight pages of hues are as follows:

Hue	Color Range
10R	reds
2.5YR	reddish browns
5YR	reddish browns and reddish yellows
7.5YR	browns and reddish yellows
10YR	browns and yellowish browns
2.5Y	yellowish browns and olive browns
5Y	yellows and olives
gley	grays, blues, and greens

On each page, the chips vary according to value and chroma. Value increases toward the top of the page (colors become lighter), and chroma increases from left to right (colors become stronger and less grey). In a full color designation, hue is given first, followed by a space, then value, then chroma. Value and chroma are separated by a forward slash; for example:

Munsell Code	Hue	Value	Chroma	Color Name
5YR 6/3	5YR	6	3	light reddish brown

Note that each color chip has a color name as well as a color code, and the color chart book includes key charts to match the color names with the chips. It is not necessary to write down the color name in the field.

Matching soils to color chips is somewhat subjective, and results will vary not only with field personnel but also with the moisture content of the soil and the type of light available. The best strategy for matching colors is first to find the closest matching hue sheet, then choose a color chip on that sheet.

4.2.1.2 Texture

Texture (grain size distribution) of the sediment will be described according to the estimated percentages of gravel, sand, silt and clay in the sample. Textural classes and the particle sizes associated with these classes are shown in Attachment 1. Descriptions of soils will be formatted according to Attachment 2, with principle component listed first followed by secondary, tertiary and so forth. A reliable field guide to determining grain size and texture in the field can be found in the W.F. McCollough sand-gauge cards.

Also to be included in the description of the texture is a description of the rock fragments in the sediment sample. These will include particle sizes over approximately 5mm in diameter. The volumetric percentage of rock fragments should be recorded. The shape of the fragments will be described according to the following terminology: rounded, subrounded, angular or subangular, as illustrated in Attachment 3. The rock type or mineral type of the fragments should also be defined whenever possible.

4.2.1.3 Consistence

The consistence of the sediment will be described whenever it remains evident in the split spoon sample. The sample may be compacted excessively or otherwise disturbed during sampling and the natural consistence may not be evident during examination. If the consistence can be characterized, it will be described according to the terms and definitions listed in Attachment 4.

The key distinction for this parameter is the change from friable to firm. When handling a sample that is friable, it will take only slight pressure between the thumb and fingers to cause a clod or ped of the sediment to break apart. A sample that is firm will resist crushing to the point where it finally seems to explode when it is subjected to adequate pressure. A friable clod or ped will not explode or pop, it will simply fall apart when pressure is applied. The other descriptive classes, as show in Attachment 4, will be applied to add more information as to the degree of soil stability, or lack thereof.

4.2.1.4 Structure

Observations of structure will include a characterization of the orientation and continuity of contacts (e.g., sharp or gradational), angularity of grains (see Attachment 3), presence of fractures, cracks, partings or macropores and a qualitative description of the degree of sorting (poorly sorted means many different grain sizes, well sorted means uniform grain size).

Additional description of such as features as crossbeds, grading, ripples and varves will aid in identifying the depositional environment.

4.2.1.5 Moisture

The soil moisture will be described on the basis of visual and manual field observations. The moisture class will be based on the percent water in the soil as follows:

Moisture Class	Moisture Content (%)	Field Description
Dry	0	No dampness is evident in sample.
Humid	1-25	Slight damp mark seen on hand when sample is squeezed.
Moist	26-75	Grading from humid to wet.
Wet	76-99	Water is released when sample is squeezed.
Saturated	100	Water drips freely from sample.

4.2.1.6 Concretions

When iron or manganese concretions are present in a soil sample, they will be noted as to their abundance and size. The use of dilute hydrochloric acid is helpful in identifying the presence of carbonate minerals, though its use is not required.

4.2.1.7 Other Parameters

The protocols or work plan for a given study may require the determination of other parameters, e.g., screening for the presence of volatile organic compounds with a photoionization detector at a hazardous waste investigation. In such cases, the protocol, work plan, additional SOPs, or other authority or regulation will describe the additional or alternate method of sediment evaluation.

4.3 Data Recording

The information required by this SOP will be recorded in a field book during the sampling process. Required information includes:

Project name

SEI study number

Site name

Well/Profile ID number

Name of geoscientist

Driller's name

Date of exploration

Attachment 5 presents a format for recording field data as well as examples of the descriptors to be used. All of the data fields must be addressed for each discrete layer being described. All measurements should be in feet and tenths of feet (not inches). The lengths of the segments of the particular sample being described must add up to the linear footage of recovery noted. In the interest of standardization, if less than complete recovery is achieved it should be assumed that the missing portion of the sample interval is at the bottom of the sample.

5.0 RESPONSIBILITIES

5.1 Principals/Group Officers/Group Leaders/Personnel

Any staff conducting these procedures are responsible for signing, dating, and managing all field observation forms.

6.0 DEFINITIONS

1. *Concretions* A nodular or irregular concentration of certain authigenic constituents of sedimentary rocks and tuffs; developed by the localized deposition of material from solution, generally about a nucleus. Harder than enclosing rock.
2. *Lamina (ae)* Unit layer or sheet of sediment in which the stratification planes are one centimeter or less apart. Laminae need not be parallel to bedding. When they are at an angle to the bedding planes the term "cross-lamination" may be used.

7.0 REFERENCES

Davis, Richard A. Jr., 1992, Depositional Systems: An Introduction to Sedimentology and Stratigraphy, 2nd Edition, Prentice Hall, New Jersey, 604 p.

Folk, R.L., Petrology of Sedimentary Rocks, Hemphill Publishing Company, 1974.

Hunter/BCA. Standard Operating Procedure No. 6006, Identification of Soils

American Geological Institute, 1976, Dictionary of Geological Terms, Revised Edition, Anchor Press/Doubleday, Garden City, New York, 472 p.

Laboratory Manual for Hand Specimen Petrology, Department of Geology, University of Texas [Undated].

Pettijohn, F.J., Sedimentary Rocks, Harper & Row, 1975.

8.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

1. Attachment 1: Wentworth Grain Size Scale
2. Attachment 2: Soil Description Format
3. Attachment 3: Angularity of Rock Fragment
4. Attachment 4: Consistence Classes
5. Attachment 5: Soil Boring Log

9.0 AUTHORIZATION

Revised by: Lee Rosberg Date: 10/16/12

Lee Rosberg, Project Scientist

Approved by: Christopher T. Stone Date: 10/16/12

Christopher T. Stone, President

10.0 REVISION HISTORY

Revision 1

1. Renumbered from Chapter SEI-10.11.1

Revision 2

1. Changed title from geologic description of unconsolidated deposits to geologic description of unconsolidated deposits using the Wentworth grain size scale
2. Added second paragraph of section 1.0
3. Expanded section 4.2.1.1 to describe color using the Munsell system
4. Updated Attachment 5 with current soil boring log
5. Several minor grammatical edits
6. Added Pettijohn (1975) and Folk (1974) references

ATTACHMENT 1

WENTWORTH GRAIN SIZE SCALE

Limiting particle diameter				Size class	
mm	phi units				
2048	-11	Very large		Boulders	GRAVEL
1024	-10	Large			
512	-9	Medium			
256	-8	Small			
128	-7	Large			
64	-6	Small		Cobbles	
32	-5	Very coarse			
16	-4	Coarse		Pebbles	
8	-3	Medium			
4	-2	Fine			
2	-1	Very fine		Granules	
1	0	Very coarse		SAND	
		µm			
1/2	1	500	Coarse		
1/4	2	250	Medium		
1/8	3	125	Fine		
1/16	4	62	Very fine		
1/32	5	31	Very coarse	Silt	MUD
1/64	6	16	Coarse		
1/128	7	8	Medium		
1/256	8	4	Fine		
1/512	9	2	Very Fine		
		<2 µm		Clay	

ATTACHMENT 2

SOIL DESCRIPTION FORMAT

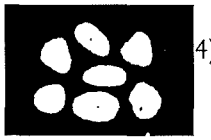
Component	Identifying Term or Format	Defining Range of Percentage by Weight
Principle	Upper case letters	Generally >50%
Secondary Component	Use adjective (ex. silty, sandy)	35% to 50%
Third Component	some	20% to 35%
	little	10% to 20%
	trace	<10%

ATTACHMENT 3
ANGULARITY OF ROCK FRAGMENTS

1) Angular: having sharp corners and edges, and therefore showing little or no effects of abrasion or wear.

2) Subangular: having edges and corners slightly rounded, so that wear is evident.

3) Subround: having most of the corners and edges worn down to smooth curves, and thus showing extensive abrasion.



4) Round: having all edges and corners smoothed off to gentle curves by prolonged wear.

ATTACHMENT 4
CONSISTENCE CLASSES

	Class	Description
Dry Soil:		
	dl —	loose- usually associated with sands
	ds —	soft- fragile
	dsh —	slightly hard- breaks easily
	dh —	hard- barely breakable with fingers
	deh —	extremely hard- can't break with fingers
Moist Soil:		
	ml —	noncoherent
	mvfr —	very friable- very easily broken apart
	mfr —	friable- easily broken apart
	mfi —	firm- resist crushing, "explode" when finally crushed
	mvfi —	very firm- barely breakable with fingers
Wet Soil:		
	wso —	nonsticky
	wss —	slightly sticky- soil sticks to one finger when squeezed and separated
	ws —	sticky- soil sticks to both thumb and finger, stretches slightly
	wvs —	very sticky- soil sticks strongly, resists separation of thumb and finger
	wsp —	slightly plastic- forms wire when rolled between hands, but breaks easily
	wp —	plastic- forms wire when rolled between hands, doesn't break easily
	wvp —	very plastic- forms a very durable wire when rolled between hands

Attachment 5

SOIL BORING LOG

Project Name: _____ Site Name: _____

Stone Project umber: Client:

BORING/WELL ID:	Date Drilled:		Location:
SOP followed:	Borehole Diam. (in)		
Deviations:	Well Diameter (in):		
Drilling Method:	Screen Length (ft):		
Driller's name:	Screen Slot Size:		
Sampling method:	Sand Pack (ft):		
Core Length:	Well Seal (ft):		
Comments:			

Depth Interval (ft bgs)	Recovery (feet)	PID		Soil Description (color, texture, moisture, remarks)	Sample Collected (Depth, ID & Time)	Well Const. Details
		Interval (ft)	Reading (ppm v/v)			

Geoscientist:	Signature:	Date:
Geoscientist:	Signature:	Date:

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Page ____ of ____

Depth Interval (ft bgs)	Recovery (feet)	PID		Soil Description (color, texture, moisture, remarks)	Sample Collected (Depth, ID & Time)	Well Const. Details
		Interval (ft)	Reading (ppm v/v)			

Geoscientist:	Signature:	Date:
Geoscientist:	Signature:	Date:

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Page ____ of ____

C: LABORATORY STANDARD OPERATING PROCEDURES

Thielsch Engineering, Inc. **SOP GT 5.0**

Standard Operating Procedure for
Total Unit Weight (Bulk Density)

Date: 9-11-12 *REV NO. 1*

Reviewed by Lab Supervisor: *Date: Sept. 12, 2012*

1. **Scope and Application**

1.1 This test method covers the determination of the specific gravity of soils that pass the 4.75mm (No. 4) sieve, by means of a pycnometer. When the soil contains particles larger than the 4.75mm sieve, Test Method C 127 shall be used for the material retained on the 4.75mm sieve and this test method shall be used for the material passing the 4.75mm sieve.

1.2 Two procedures for performing the specific gravity are provided as follows:

1.2.1 Method A - Procedure for Oven-Dry Specimens.

1.2.2 Method B - Procedure for Moist Specimens. The procedure to be used shall be specified by the requesting authority. For specimens of organic soils the highly plastic, fine grained soils, Procedure B Shall be the preferred method.

2. **Method Summary**

2.1 Related Standard Operating Procedures - None

3. **Sample Preservation, Containers, Handling and Storage**

Preservation: Non-Applicable

Containers: Jars - glass or plastic (4 - 32 oz.), plastic sample bags (5 gallon), 5 gallon buckets.

Storage: Samples are stored in Laboratory until project is complete, data is sent and no additional testing is required.

4. **Interference and Potential Problems - Loss of sample during weighing and measuring dimensions.**

- Sample size is sometimes compromised due to limited amount of material to perform analysis.
- Organic matter can float causing inaccurate meniscus readings.
- Error in recording of weights during any step of this analysis.
- Error in calculating data.

5. **Definitions**

5.1 Specific Gravity: The ratio of the mass of a unit volume of a material at a stated temperature to the mass of the same volume of gas-free distilled water at a stated temperature.

6. **Equipment and Apparatus**

6.1 Pycnometer - The pycnometer shall be one of the following:

6.1.1 Volumetric flask having a capacity of at least 100 mL.

6.1.2 Stoppered bottle having a capacity of at least 50mL. The stopper shall be of the same material and shall permit the emission of air and surplus water when it is put in place.

6.2 Balance - Meeting the requirements of Specification D 4753 and readable, without estimation, to at least 0.1% of the specimen mass.

6.3 Drying Oven - Thermostatically-controlled oven, capable of maintaining a uniform temperature of 110±°C (230± 9 °F) throughout the drying chamber.

6.4 Thermometer, capable of measuring the temperature range within which the test is being performed, graduated in a 0.5°C (1.0°F) division scale.

6.5 Entrapped Air Removal Apparatus - To remove entrapped air, use on or a combination of the following:

6.5.1 Hot plate or Bunsen burner, capable of maintaining a temperature adequate to boil water.

6.5.2 Vacuum system, a vacuum pump or water aspirator, capable of producing a partial vacuum of 26 In Hg.

6.6 Miscellaneous Equipment, specimen dishes and insulated gloves

7. **Reagents**

- Distilled and/or demineralized water

8. **Health and Safety Issues**

Safety glasses, long pants, socks, gloves and laboratory coats must be worn when handling samples with known contaminants.

The Laboratory Manager ensures that health and safety (H&S) procedures are discussed with each new employee by the H&S representative prior to beginning work in the laboratory.

The chemical hygiene plan and the Hazardous Materials Handling and Disposal SOP should be reviewed by each employee. Copies of both plans are kept in the laboratory.

Use caution when dealing with any heated oven zones. Wear heat protection gloves.

9. **Procedures**

9.1 Procedures
Login:
Samples are logged in as follows:

- Each sample is given a unique laboratory identification number.
- This number must be sequential beginning with number one.
- In the case of ongoing project, refer to next Lab No. in numerical sequence.
- Last Laboratory No. can be found in log book in laboratory.
- This log must be updated each time a new sample is logged in.

9.2 Identify Sample for Testing: Check soils laboratory testing assignment.

9.3 Specific Gravity test is assigned if an X is marked under Gs column of Identification Tests and Sample Information (Boring / Test Pit No., Depth,) coincides with sample information on sample container.

9.4 Sample Preparation and Procedure

- Take entire sample and screen through a #4 sieve. After screening, take all minus #4 material, mix thoroughly and take a representative portion (approximately 30 - 50 grams dry weight).

  Note: Representative portion is obtained by either decanting through a sample splitter or using the quartering method.

- Put sample into a calibrated 500mL pycnometer using funnel and add distilled or demineralized water to below measured line on pycnometer.
- Place pycnometer in a warm temperature bath (30° - 38° C) and connect vacuum line using rubber stoppered bottle cap to top of pycnometer.
- Apply vacuum (minimum 26 reading on vacuum gage) to sample and allow all entrapped air to escape. Apply vacuum for at least 30 minutes.
- Shake pycnometer slightly with hand and determine visually that no additional air is present in sample. If air is present, continue vacuum until such time that shaking pycnometer produces no entrapped air.
- Remove vacuum from open end of pycnometer and take pycnometer out of warm temperature bath.
- With an eyedropper, adjust water level to desired line on pycnometer.
- Wipe outside of pycnometer clean with paper Towel to remove any moisture and/or other particles.
- Record weight of pycnometer (wt. of bottle + water + soil in grams) under Determination No. 1.

- Immediately after recording weight, insert thermometer and record temperature (temperature, T. in °C) to the nearest .1°C, again under Determination No. 1.

- Repeat prior step up to 3 times at various temperature reading, if meniscus is hard to read , 4 determinations may be required.

- Weigh rectangular pan (capable of containing approximately 800 grams of material) and record as Wt. Dish in grams.

- Take pycnometer and agitate by turning and swirling in a circular motion with hand.

- Begin decanting soil/water mix pycnometer into rectangular pan.

- Take squirt bottle and rinse inside of pycnometer to get remaining particles into suspension and again decant into pan.

- Repeat prior step until no visual soil remains in pycnometer.

- Place pan and soil in oven and dry for not less than 16 hours at 110°C ± 5°C.
  Note: Due to large amount of water, sample may need additional drying time.

- Remove pan from water and allow to cool.

- After cooling, weigh pan and record under Wt of Dish & Dry Soil.

9.5 Status Check on Data:
Status is obtained from the soils laboratory testing assignment sheet as follows:

- Test assigned is clearly designated with an X in appropriate column (Step 9.3).

- If one quarter of X is shaded, sample has been prepared.

- If one half of X is shaded, sample has been run.

- If three quarters of X is shaded, test has been calculated.

- Finally, if entire X is shaded, test has been reduced, reviewed and can be sent as final data.

9.6 Data Reduction: All data upon conclusion of test is then input into a Microsoft Excel spreadsheet and furnished along with raw data to laboratory director or supervisor for data review.

9.7 Data Review: Data Review is done as follows:

- Check calculation for weight of sample in grams.

- Check specific gravity versus soil description.

- After specific gravity determination has been verified, cross-examine job name, sample identification, soil classification and any other pertinent information.

10. **Calculations: Volume, Total Unit Weight**

10.1Volume

$$V\ (ft^3) = \left[\frac{\Pi D^2}{4}(L_o)\right] \div 1728$$

II = 3.14
D = Diameter
1728 - Conversion of in^3 to ft^3 (i.e. 12X12X12)
L_0 = Initial length

10.2Total Unit Weight (Bulk Density)

Total Unit WT (pcf) = $\left[\frac{WT\ of\ Wet\ Sample}{V}\right]$ ÷ 453.59 grams

Where V = Volume in ft^3

453.59 = conversion of grams to pounds $\left(\frac{1\ lb.}{453.59\ gms.}\right)$

11. **Calibration**

- Balances are checked daily with S-class weights and kept in Log Book in laboratory by scale (SOP SA 3.0).

12. **QC Limits**

Non-Applicable

13. **Corrective Action Procedures**

13.1Corrective actions are taken as follows:

13.1.1 Any suspect or questionable data during test is to be reported to Supervisor immediately on finding

13.1.2 Supervisor will review data and check any calculations for error

13.1.3 If there is a calculation error that is made, correction will be noted and test will continue.

13.1.4 If error cannot be retrieved, then Supervisor instructs technician to retrieve any unused portion of sample.

13.1.5 When unused sample is retrieved, Supervisor determines if there is enough material and if so instructs technician to re-run entire test. If there is not enough material to re-run test, Supervisor relays that information to Project Manager or client. At this stage, it is determined whether more sample can be obtained to run analysis or test is canceled.

13.1.6 During data review if there are any errors or questionable data. Laboratory Director notifies Supervisor and Supervisor retrieves test sample. If checking test sample does not correct mistake then refer to 13.1.5.

13.1.7 Any corrections made to data sheet are done by putting a line through error and recording new data and initialing by individual. If more than one mistake is made and there is no room on data sheet to put correction, a line will be placed through error, a new data sheet will be stapled to original data and a brief explanation as to why sheet is stapled, then new data recorded on staples sheet and initialed by individual.

13.1.8 Any changes, deviances or unusual measures taken during recording of data shall have an asterisk placed next to unusual data and a brief yet detailed description of what data represents and why it is represented in this manner, followed by initials of individual taking these actions.

14. **Document Description and Example Forms**

14.1 See attached forms.

15. **Miscellaneous Notes**

None

16. **References**

ASTM D1587 - Thin Walled Tube Geotechnical Sampling of Soils

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Thielsch Engineering, Inc. **SOP GT 5.0**

Standard Operating Procedure for
Standard Test Method for Specific Gravity of Soils

Date: 9-11-12 *REV NO. 1*

Reviewed by Lab Supervisor: *Date: Sept. 12, 2012*

1. **Scope and Application**

1.1 This test method covers the determination of the specific gravity of soils that pass the 4.75mm (No. 4) sieve, by means of a pycnometer. When the soil contains particles larger than the 4.75mm sieve, Test Method C 127 shall be used for the material retained on the 4.75mm sieve and this test method shall be used for the material passing the 4.75mm sieve.

1.2 Two procedures for performing the specific gravity are provided as follows:

1.2.1 Method A - Procedure for Oven-Dry Specimens.

1.2.2 Method B - Procedure for Moist Specimens. The procedure to be used shall be specified by the requesting authority. For specimens of organic soils the highly plastic, fine grained soils, Procedure B Shall be the preferred method.

2. **Method Summary**

2.1 Related Standard Operating Procedures - None

3. **Sample Preservation, Containers, Handling and Storage**

Preservation: Non-Applicable

Containers: Jars - glass or plastic (4 - 32 oz.), plastic sample bags (5 gallon), 5 gallon buckets.

Storage: Samples are stored in Laboratory until project is complete, data is sent and no additional testing is required.

4. **Interference and Potential Problems - Loss of sample during weighing and measuring dimensions.**

- Sample size is sometimes compromised due to limited amount of material to perform analysis.
- Organic matter can float causing inaccurate meniscus readings.
- Error in recording of weights during any step of this analysis.
- Error in calculating data.

5. **Definitions**

5.1 Specific Gravity: The ratio of the mass of a unit volume of a material at a stated temperature to the mass of the same volume of gas-free distilled water at a stated temperature.

6. **Equipment and Apparatus**

6.1 Pycnometer - The pycnometer shall be one of the following:

6.1.1 Volumetric flask having a capacity of at least 100 mL.

6.1.2 Stoppered bottle having a capacity of at least 50mL. The stopper shall be of the same material and shall permit the emission of air and surplus water when it is put in place.

6.2 Balance - Meeting the requirements of Specification D 4753 and readable, without estimation, to at least 0.1% of the specimen mass.

6.3 Drying Oven - Thermostatically-controlled oven, capable of maintaining a uniform temperature of 110±°C (230± 9 °F) throughout the drying chamber.

6.4 Thermometer, capable of measuring the temperature range within which the test is being performed, graduated in a 0.5°C (1.0°F) division scale.

6.5 Entrapped Air Removal Apparatus - To remove entrapped air, use on or a combination of the following:

6.5.1 Hot plate or Bunsen burner, capable of maintaining a temperature adequate to boil water.

6.5.2 Vacuum system, a vacuum pump or water aspirator, capable of producing a partial vacuum of 26 In Hg.

6.6 Miscellaneous Equipment, specimen dishes and insulated gloves

7. **Reagents**

- Distilled and/or demineralized water

8. **Health and Safety Issues**

Safety glasses, long pants, socks, gloves and laboratory coats must be worn when handling samples with known contaminants.

The Laboratory Manager ensures that health and safety (H&S) procedures are discussed with each new employee by the H&S representative prior to beginning work in the laboratory.

The chemical hygiene plan and the Hazardous Materials Handling and Disposal SOP should be reviewed by each employee. Copies of both plans are kept in the laboratory.

Use caution when dealing with any heated oven zones. Wear heat protection gloves.

9. **Procedures**

9.1 Procedures
Login:
Samples are logged in as follows:

- Each sample is given a unique laboratory identification number.
- This number must be sequential beginning with number one.
- In the case of ongoing project, refer to next Lab No. in numerical sequence.
- Last Laboratory No. can be found in log book in laboratory.
- This log must be updated each time a new sample is logged in.

9.2 Identify Sample for Testing: Check soils laboratory testing assignment.

9.3 Specific Gravity test is assigned if an X is marked under Gs column of Identification Tests and Sample Information (Boring / Test Pit No., Depth,) coincides with sample information on sample container.

9.4 Sample Preparation and Procedure

- Take entire sample and screen through a #4 sieve. After screening, take all minus #4 material, mix thoroughly and take a representative portion (approximately 30 - 50 grams dry weight).

  Note: Representative portion is obtained by either decanting through a sample splitter or using the quartering method.

- Put sample into a calibrated 500mL pycnometer using funnel and add distilled or demineralized water to below measured line on pycnometer.
- Place pycnometer in a warm temperature bath (30°C - 38°C) and connect vacuum line using rubber stoppered bottle cap to top of pycnometer.
- Apply vacuum (minimum 26 reading on vacuum gage) to sample and allow all entrapped air to escape. Apply vacuum for at least 30 minutes.
- Shake pycnometer slightly with hand and determine visually that no additional air is present in sample. If air is present, continue vacuum until such time that shaking pycnometer produces no entrapped air.
- Remove vacuum from open end of pycnometer and take pycnometer out of warm temperature bath.
- With an eyedropper, adjust water level to desired line on pycnometer.
- Wipe outside of pycnometer clean with paper Towel to remove any moisture and/or other particles.
- Record weight of pycnometer (weight of bottle + water + soil in grams) under Determination No. 1.

- Immediately after recording weight, insert thermometer and record temperature (temperature, T. in °C) to the nearest .1°C, again under Determination No. 1.

- Repeat prior step up to 3 times at various temperature readings, if meniscus is hard to read, 4 determinations may be required.

- Weigh rectangular pan (capable of containing approximately 800 grams of material) and record as Wt. Dish in grams.

- Take pycnometer and agitate by turning and swirling in a circular motion with hand.

- Begin decanting soil/water mix pycnometer into rectangular pan.

- Take squirt bottle and rinse inside of pycnometer to get remaining particles into suspension and again decant into pan.

- Repeat prior step until no visual soil remains in pycnometer.

- Place pan and soil in oven and dry for not less than 16 hours at 110°C ± 5°C.

  Note: Due to large amount of water, sample may need additional drying time.

- Remove pan from water and allow to cool.

- After cooling, weigh pan and record under Wt. of Dish & Dry Soil.

9.5 Status Check on Data:

Status is obtained from the soils laboratory testing assignment sheet as follows:

- Test assigned is clearly designated with an X in appropriate column (Step 9.3).

- If one quarter of X is shaded, sample has been prepared.

- If one half of X is shaded, sample has been run.

- If three quarters of X is shaded, test has been calculated.

- Finally, if entire X is shaded, test has been reduced, reviewed and can be sent as final data.

9.6 Data Reduction: All data, upon conclusion of test is then hand calculated and input onto a Microsoft Excel spreadsheet and furnished along with raw data to laboratory director or supervisor for data review.

9.7 Data Review: Data Review is done as follows:

- Check calculation for weight of sample in grams.

- Check specific gravity versus soil description.

- After specific gravity determination has been verified, cross-examine job name, sample identification, soil classification and any other pertinent information.

9.8 Data Report:

Final data is sent as follows:

- Identification of the sample (material) being tested such as boring number, sample number, test number, etc.
- Specific gravity at 20°C to the nearest 0.01.

10. Calculations: Volume, Total Unit Weight

10.1 Formula

$$Gs = \left[\frac{GtWs}{Ws - W1 + W2}\right]$$

Where:

Gs = Specific Gravity

Gt = Density of water at given temperature (see Table 1 ASTM D584)

Ws = Weight of Soil in grams

W1 = Weight of pycnometer, water, and soil in grams

W2 = Weight of bottle and water in grams (see calibration folder for pycnometer used)

10.2 Verification of Calculations

- Review all determinations to insure the Gs values are all similar.
- Check soil description and any other test data associated with sample to see if Gs correlates with other test values.

11. Calibration

- Oven temperature is recorded daily and calibrated every four months (SOP SA-Draft)
- Balances are checked daily with S class weights and kept in log book in laboratory by Scale (SOP SA 3.0)
- Pycnometer are calculated annually (SOP Draft)

12. QC Limits

Non-Applicable

13. Corrective Action Procedures

13.1 Corrective actions are taken as follows:

13.1.1 Any suspect or questionable data during test is to be reported to Supervisor immediately on finding

13.1.2 Supervisor will review data and check any calculations for error

13.1.3 If there is a calculation error that is made, correction will be noted and test will continue.

13.1.4 If error cannot be retrieved, then Supervisor instructs technician to retrieve any unused portion of sample.

13.1.5 When unused sample is retrieved, Supervisor determines if there is enough material and if so instructs technician to re-run entire test. If there is not enough material to re-run test, Supervisor relays that information to Project Manager or client. At this stage, it is determined whether more sample can be obtained to run analysis or test is canceled.

13.1.6 Supervisor or laboratory technician appointed by Supervisor must weigh and calculate all oven-dried samples every morning. This is a routine check of testing to determine any errors in recording of weights during test.

13.1.7 When an error occurs during 13.1.6 the following measure is taken: first re-weigh sample and container and check against recorded weight. If weight does not check, then re-record new weight and re-calculate to see if new data correlates with all other data. If weight checks, then remove oven dried material an weigh container and repeat prior step. If correction in made, the test can continue or be reduced and reviewed. If a correction is not made then refer to 13.1.5 for corrective action.

13.1.8 Oven-dried samples are not disposed of until Supervisor has checked all data calculations and determines that data is valid.

13.1.9 During data review if there are any errors or questionable data Laboratory Director notifies Supervisor and Supervisor retrieves test sample. If checking test sample by weighing or sieving corrects error then test is reduced again and sent through for review. If checking test sample does not correct mistake, refer to 13.1.5.

13.1.10 Any corrections made to data sheet are done by putting a line through error and recording new data and initialing by individual. If more than one mistake is made and there is no room on data sheet to put correction, a line will be placed through error, a new data sheet will be stapled to original data and a brief explanation as to why sheet is stapled, then new data recorded on staples sheet and initialed by individual.

13.1.11 Any changes, deviances or unusual measures taken during recording of data shall have an asterisk placed next to unusual data and a brief yet detailed description of what data represents and why it is represented in this manner, followed by initials of individual taking these actions.

14. **Document Description and Example Forms**

14.1 See attached forms.

15. **Miscellaneous Notes**

None

16.**References**

ASTM D853 - Standard Test Method for Specific Gravity of Soils

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Thielsch Engineering, Inc. **SOP GT 7.0**

Standard Operating Procedure for Moisture, Ash and Organic Matter of Peat and Other Organic Soils

Date: 9-11-12 *REV NO. 1*

Reviewed by Lab Supervisor: *Date: Sept. 12, 2012*

1. **Scope and Application**

1.1 These test methods cover the measurement of moisture content, ash content and organic matter in peats and other organic soils, such as organic clays, silts and mucks.

1.2 Two procedures for performing the specific gravity are provided as follows:

1.2.1Method A - Procedure for Oven-Dry Specimens.

1.2.2 Method B - Procedure for Moist Specimens. The procedure to be used shall be specified by the requesting authority. For specimens of organic soils the highly plastic, fine grained soils, Procedure B Shall be the preferred method.

2. **Method Summary**

2.1 Related Standard Operating Procedures

2.2 Test Method A: Moisture is determined by drying a peat or organic soil sample at 105°C. T. The moisture content is expressed either as a percent of the oven-dry mass or of the as-received mass.

2.3 Test Method B: Ash content of a peat or organic soil sample is determined by igniting the oven-dried sample from the moisture content determination in a muffle furnace at 440°C (Method C). The substance remaining after ignition is the ash. The ash content is expressed as a percentage of the mass of the oven-dried sample.

2.4 Organic Matter: Organic matter is determined by subtracting percent ash content from one hundred.

3. **Equipment and Apparatus**

3.1 Oven, capable of being regulated to a constant temperature of 105±5°C.

3.2 Muffle furnace, capable of producing constant temperature of 440°C.

3.3 Evaporating dishes, of high silica or porcelain of not less than100mL capacity.

3.4 Porcelain pan, spoons and equipment of like.

3.5 Dessicator.

4. **Interference and Potential Problems**

5. **Definitions**

6. **Equipment and Apparatus**

7. **Reagents**

8. **Health and Safety Issues**

The Laboratory Manager ensures that health and safety (H&S) procedures are discussed with each new employee by the H&S representative (or designee) prior to beginning work in the laboratory.

All samples are to be treated as though they are hazardous. Work involving standards and solvents must be performed under a fume hood.

Safety glasses are to be worn when working with samples, solvents, standards and while in the vicinity of others working with chemicals. Long pants, socks, gloves and laboratory coats must be worn when handling samples and extracts.

The chemical hygiene plan and the Hazardous Materials Handling and Disposal SOP should be reviewed by each employee. Copies of both plans are kept in the laboratory.

9. **Procedures**

9.1 Procedures

Login:

Samples are logged in as follows:

- Each sample is given a unique laboratory identification number.
- This number must be sequential beginning with number one.
- In the case of ongoing project, refer to next Lab No. in numerical sequence.
- Last Laboratory No. can be found in log book in laboratory.
- This log must be updated each time a new sample is logged in.

10. **Calculations: Volume, Total Unit Weight**

10.1Formula

Moisture Content = 100 (wt. wet soil & cup - wt. dry soil & cup) / (wt. dry soil & cup - wt. cup)

Ash % = 100 (wt. ash & cup - wt. cup) / (wt. dry soil & cup - wt. cup)

Organic % = 100 - Ash %

10.2 Verification of Calculations

11. Data Validation

12. QC Limits

Non-Applicable

13. Corrective Action Procedures

13.1 Corrective actions are taken as follows:

13.1.1 Any suspect or questionable data during test is to be reported to Supervisor immediately on finding

13.1.2 Supervisor will review data and check any calculations for error

13.1.3 If there is a calculation error that is made, correction will be noted and test will continue.

13.1.4 If error cannot be retrieved, then Supervisor instructs technician to retrieve any unused portion of sample.

13.1.5 When unused sample is retrieved, Supervisor determines if there is enough material and if so instructs technician to re-run entire test. If there is not enough material to re-run test, Supervisor relays that information to Project Manager or client. At this stage, it is determined whether more sample can be obtained to run analysis or test is canceled.

13.1.6 Supervisor or laboratory technician appointed by Supervisor must weigh and calculate all oven-dried samples every morning. This is a routine check of testing to determine any errors in recording of weights during test.

13.1.7 When an error occurs during 13.1.6 the following measure is taken: first re-weigh sample and container and check against recorded weight. If weight does not check, then re-record new weight and re-calculate to see if new data correlates with all other data. If weight checks, then remove oven dried material and weigh container and repeat prior step. If correction is made, test can continue or be reduced and reviewed. If correction is not made then refer to 13.1.5 for corrective action.

13.1.8 Oven-dried samples are not disposed of until Supervisor has checked all data calculations and determines data is valid.

13.1.9 During data review if there are any errors or questionable data Laboratory Director notifies Supervisor and Supervisor retrieves test sample. If checking test sample by weighing or sieving corrects error then test is reduced again and sent through for review. If checking test sample does not correct mistake, refer to 13.1.5.

13.1.10 Any corrections made to data sheet are done by putting a line through error and recording new data and initialing by individual. If more than one mistake is made and there is no room on data sheet to put correction, a line will be placed through error, a new data sheet will be stapled to original data and a brief explanation as to why sheet is stapled, and then new data recorded on staples sheet and initialed by individual.

13.1.11 Any changes, deviances or unusual measures taken during recording of data shall have an asterisk placed next to unusual data and a brief yet detailed description of what data represents and why it is represented in this manner, followed by initials of individual taking these actions.

14. **Document Description and Example Forms**

15. **Miscellaneous Notes**

None

16. **References**

ASTMD2974 - Standard Test Method for Moisture, Ash and Organic Matter of Peat and Other Organic Soils

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ESS Laboratory
Division of Thielsch Engineering
Cranston, RI

SOP NO. 20_5035
Volatile Organic Compounds
by Gas Chromatography/Mass Spectrometry (GC/MS):
Capillary Column Technique
(SW-846 METHOD 5035)

APPROVED BY:

Operations Manager	Date 3/13/09
QA Manager	Date 3/16/09
Laboratory Director	Date 3/13/09

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Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS): Capillary Column Technique (SW-846 METHOD 5035)

1.0 SCOPE AND APPLICATION

1.1 This method describes the technique utilized by ESS Laboratory in the qualitative and quantitative analysis of volatile samples based on SW-846 Method 5035. Following are compounds that are currently determined by this method.

Appropriate Technique Analyte	Cas #[b]	Purge-and-Trap	Direct Injection
Acetone	67-64-1	Pp	a
Acrolein (Propenal)	107-02-8	Pp	a
Acrylonitrile	107-13-1	Pp	a
Allyl chloride	107-05-1	a	a
tert- Amyl methyl ether	994-05-8	a	a
Benzene	71-43-2	a	a
Bromobenzene	108-86-1	a	a
Bromochloromethane	74-97-5	a	a
Bromodichloromethane	75-27-4	a	a
4-Bromofluorobenzene	460-00-4	a	a
Bromoform	75-25-2	a	a
Bromomethane	74-83-9	a	a
2-Butanone (MEK)	78-93-3	Pp	a
n-Butylbenzene	104-51-8	a	a
sec-Butylbenzene	135-98-8	a	a
tert-Butylbenzene	98-06-6	a	a
Carbon disulfide	75-15-0	Pp	a
Carbon tetrachloride	56-23-5	a	a
Chlorobenzene	108-90-7	a	a
1-Chlorobutane	109-69-3	a	a
Chlorodibromomethane	124-48-1	a	a
Chloroethane	75-00-3	a	a
2-Chloroethyl vinyl ether	110-75-8	a	a
Chloroform	67-66-3	a	a

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Chloromethane	74-87-3	a	a
Chloroprene	126-99-8	a	a

Appropriate Technique Analyte	Cas #[b]	Purge-and-Trap	Direct Injection
2-Chlorotoluene	95-49-8	a	a
4-Chlorotoluene	106-43-4	a	a
Cyclohexane	110-82-7	a	a
1,2-Dibromo-3-chloropropane	96-12-8	Pp	a
1,2-Dibromoethane	106-93-4	a	a
Dibromomethane	74-95-3	a	a
1,2-Dichlorobenzene	95-50-1	a	a
1,3-Dichlorobenzene	541-73-1	a	a
1,4-Dichlorobenzene	106-46-7	a	a
Cis-1,4-Dichloro-2-butene	1476-11-5	a	a
trans-1,4-Dichloro-2-butene	110-57-6	Pp	a
Dichlorodifluoromethane	75-71-8	a	a
1,1-Dichloroethane	75-34-3	a	a
1,2-Dichloroethane	107-06-2	a	a
1,1-Dichloroethene	75-35-4	a	a
cis-1,2-Dichloroethene	156-59-2	a	a
trans-1,2-Dichloroethene	156-60-5	a	a
1,2-Dichloropropane	78-87-5	a	a
1,3-Dichloropropane	142-28-9	a	a
2,2-Dichloropropane	594-20-7	a	a
1,1-Dichloropropene	563-58-6	a	a
cis-1,3-Dichloropropene	10061-01-5	a	a
trans-1,3-Dichloropropene	10061-02-6	a	a
1,4-Difluorobenzene	540-36-3	a	a
Diethyl Ether	60-29-7	a	a
1,4-Dioxane	123-91-1	Pp	a
Ethylbenzene	100-41-4	a	A
Ethyl methacrylate	97-63-2	a	a
Ethyl tertiary-butyl ether	637-92-3	a	a
Hexachlorobutadiene	87-68-3	a	a
Hexachloroethane	67-72-1	i	a

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2-Hexanone	591-78-6	Pp	a

Appropriate Technique Analyte	Cas #[b]	Purge-and-Trap	Direct Injection
Iodomethane	74-88-4	a	a
Isopropylbenzene	98-82-8	a	a
Di-isopropyl ether	108-20-3	a	a
p-Isopropyltoluene	99-87-6	a	a
Methyl acetate	79-20-9	a	a
Methyl acrylate	96-33-3	a	a
Methylacrylonitrile	126-98-7	pp	a
Methyl Cyclohexane	108-87-2	a	a
Methylene chloride	75-09-2	a	a
Methyl methacrylate	80-62-6	a	a
4-Methyl-2-pentanone (MIBK)	108-10-1	pp	a
Naphthalene	91-20-3	a	a
2-Nitropropane	79-46-9	a	a
Pentachloroethane	76-01-7	i	a
n-Propylbenzene	103-65-1	a	a
Styrene	100-42-5	a	a
1,1,1,2-Tetrachloroethane	630-20-6	a	a
1,1,2,2-Tetrachloroethane	79-34-5	a	a
Tetrachloroethene	127-18-4	a	a
Toluene	108-88-3	a	a
1,2,3-Trichlorobenzene	87-61-6	a	a
1,2,4-Trichlorobenzene	120-82-1	a	a
1,3,5-Trichlorobenzene	108-70-3	a	a
1,1,1-Trichloroethane	71-55-6	a	a
1,1,2-Trichloroethane	79-00-5	a	a
Trichloroethene	79-01-6	a	a
Trichlorofluoromethane	75-69-4	a	a
1,2,3-Trichloropropane	96-18-4	a	a
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	a	a
1,2,4-Trimethylbenzene	95-63-6	a	a
1,3,5-Trimethylbenzene	108-67-8	a	a
Vinyl acetate	108-05-4	a	a
Vinyl chloride	75-01-4	a	a
o-Xylene	95-47-6	a	a
m-Xylene	108-38-3	a	a

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p-Xylene	106-42-3	a	a

a Adequate response by this technique.
b Chemical Abstract Services Registry Number.
i Inappropriate technique for this method
pp Poor purging efficiency results in high MRLs.

1.2 This method is used to determine volatile organic compounds in a solid material. This includes soils, sediments, solid waste and oil samples.

1.3 This method can be used to quantify most volatile organic compounds that have boiling points below 200°C and that are insoluble or slightly soluble in water. Volatile water-soluble compounds can be included in this analytical technique, however, for the more soluble compounds quantitation limits are approximately ten times higher because of poor purging efficiency.

1.4 The quantitation limit of this method for an individual compound is approximately 50 µg/Kg and 5 µg/Kg for the high and low soil/sediment methods, respectively, at 100% solids. The Quantitation limit for aqueous samples is listed in SOP 20_8260B.

2.0 METHOD SUMMARY

2.1 This SOP provides gas chromatographic/mass spectrometric (GC/MS) conditions for the detection of volatile organic compounds.

2.2 This SOP describes a closed system purge and trap procedure for soil/sediment samples.

2.3 This SOP only addresses sample analysis by purge-and-trap according to 5035 with method 8260B as the determinative method.

2.3.1 Purged sample components are trapped in a tube containing suitable sorbent materials.

2.3.2 When purging is complete, the sorbent tube is heated and back-flushed with helium to desorb trapped sample components. The analytes are desorbed directly to a capillary column for analysis.

2.3.3 Wide-bore capillary columns require a jet separator, whereas narrow bore capillary columns can be directly interfaced to the ion source.

2.4 A temperature program is used in the gas chromatograph to separate the organic compounds. Detection is achieved using a mass spectrometer (MS).

2.5 Tentative identifications are obtained by analyzing standards under the same conditions used for samples and comparing resultant GC retention times. Absolute identifications are obtained by comparing the mass spectra of individual compounds to the reference spectra for that compound.

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2.6 Concentrations of the identified components are measured by relating the response produced for that compound to the response produced by a compound that is used as an internal standard.

2.7 Soil samples with a significant moisture content (>10%) that are extracted prior to analysis in a water miscible solvent such as methanol are diluted by the total volume of the solvent/water mixture. There is the potential to underestimate the volatile content of the soil if this ***solvent dilution effect*** is not taken into account. The total mixture volume can only be calculated based on the sample moisture as determined by the % moisture. Total volume is then expressed as V_t in the sample concentration calculation provided in 9.6.1, ESS Laboratory corrects for the solvent dilution effect for all soil/sludge samples. This total solvent/water volume is calculated as follows:

$$\mu L \text{ solvent/water } V_t = \frac{[\text{ml of MeOH} + (\%\text{ moisture x g sample})]}{100} \times 1000\ \mu L/ml$$

2.8 Refer to SOP 10.0001, Attachment G, for subsampling procedures.

3.0 HEALTH AND SAFETY

3.1 Each employee has been trained and has acknowledged being trained in the safe use and handling of chemicals being used in the laboratory. This training has been performed according to the ESS Training SOP 80_0016 and by the Chemical Hygiene Plan SOP 90_0001 in conjunction with the Safety orientation

3.2 All sample and material handling should be done in a hood while using proper protective equipment to minimize exposure to liquid or vapor. Minimum personnel protective equipment includes the use of laboratory safety glasses, a lab coat or apron, and protective gloves.

3.3 The MSDSs for the concentrated chemicals used in the laboratory are kept on file in a central location that is available for all employees to review.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

4.1 **Low Level Method**: Sample vials will be provided by the laboratory and will contain 1 magnetic stir bar and 5 ml de-ionized water. A minimum of 2 vials must be submitted per sample. Vials will be pre-weighed by the laboratory (see bottle prep procedure Attachments C and D). Before GC/MS analysis, the sample vial will be re-weighed and the final soil weight will be calculated. Samples must be received within 48 hours of collection and frozen at receipt (<7°C). Store sample vials at a slant to avoid breaking vials.

4.2 **High Level Method**: Sample vials will be provided by the laboratory and will contain 15ml of methanol (purge and trap grade) as preservative. Vials will be pre-weighed by the laboratory and a scribed mark will be made indicating the level the methanol should be when 15g of soil is added to the container. Before GC/MS analysis, the sample vial will be re-weighed and the final soil weight will be calculated (see Attachments E and F). Samples extracted in Methanol are stored in a refrigerator at 4 ± 2°C within the laboratory.

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4.3 **The Encore® sample collection, storage and preparation procedure** is described in Attachment J.

4.4 Samples for analysis using this SOP must be stored in tightly sealed vials with Teflon-lined silicone septum seals in which the original sample was collected. See sample storage SOP 10_0002.

4.5 Samples known to contain high concentrations of VOCs are stored in a separate refrigerator located in the sample storage room. This is to prevent cross contamination.

4.6 Samples for volatile analysis must be analyzed within 14 days of sample collection.

4.7 Glassware is prepared for use by placing in 105°C oven overnight to ensure that there is no solvent contamination.

4.8 Low-level method trip blanks are prepared with 5ml organic free water. This water comes from a carbon filtration system located in the VOA lab and is periodically checked for low level VOCs. High-level method trip blanks are prepared using 15ml of purge and trap grade methanol.

4.9 A trip blank must accompany each batch of 20 or less VOC samples. This trip blank must accompany the associated samples at all times.

4.10 Refrigerator blanks are prepared like trip blanks and stored in the refrigerator with samples. These blanks are analyzed weekly to check for possible cross contamination from samples. To prevent cross contamination from sample to sample, all sample containers must be airtight. If the blank is greater than the MRL, then notify operations manager immediately.

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

5.1 Major sources of contamination are volatile materials in the laboratory and impurities in the inert purging gas and in the sorbent trap. The use of non-polytetrafluoroethylene (PTFE) thread sealant, plastic tubing, or flow controllers with rubber components should be avoided since such materials out-gas organic compounds which will be concentrated in the trap during the purge operation.

5.2 Analyses of reagent blanks provide information about the presence of contaminants. When potential interfering peaks are noted in blanks, the analyst should change the purge gas source. Subtracting blank values from sample results **is not** permitted.

5.3 Interfering contamination may occur when a sample containing low concentrations of volatile organic compounds is analyzed immediately after a sample containing high concentrations of volatile organic compounds. After analysis of a sample containing high concentrations of volatile organic compounds, one or more instrument blanks must be analyzed to check for cross contamination.

5.3.1 This interference may be prevented by rinsing the purging apparatus and sample syringes with portions of organic-free reagent water between samples.

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5.3.2 In extreme situations, the whole purge and trap device may require dismantling and cleaning.

5.4 Special precautions must be taken to avoid contamination when analyzing for methylene chloride. The analytical and sample storage area must be isolated from all atmospheric sources of methylene chloride. Otherwise, random background levels will result. Since methylene chloride will permeate through PTFE tubing, all gas chromatography carrier gas lines and purge gas plumbing must be constructed from stainless steel or copper tubing. Laboratory clothing worn by the analyst should be clean since clothing previously exposed to methylene chloride fumes during liquid/liquid extraction procedures can contribute to sample contamination.

5.5 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal into the sample during shipment and storage. A trip blank prepared from organic-free reagent water and carried through the sampling and handling protocol can serve as a check on such contamination.

5.6 This procedure can be used to quantitate most volatile organic compounds that have boiling points below 200°C and that are insoluble or slightly soluble in water. Volatile water-soluble compounds can be included in this analytical technique. However, for the more soluble compounds, quantitation limits are approximately ten times higher because of poor purging efficiency. Such compounds include low-molecular-weight-halogenated hydrocarbons, aromatics, ketones, nitriles, acetates, acrylates, ethers and sulfides.

6.0 EQUIPMENT/APPARATUS

6.1 **Microsyringes**: 10 µL, 25 µL, 50 µL, 100 µL, 250 µL, 500 µL, and 1,000 µL. These syringes should be equipped with a 20 gage (0.006" ID) needle.

6.2 **Disposable micropipettes**: 10, 50, 100 and 200 µL volumes. Purchased from Drummond.

6.3 **Syringe**: 5 ml and 25 ml glass, gas-tight, with Luerlock tip.

6.4 **Balance**: Top-loading balance capable of weighing 0.1 g.

6.5 **Micro-reaction vessels**: 1.0 ml and 5.0 ml purchased from Supelco. Catalogue No. 3-3293 with Mininert Caps. Clean reaction vessels by placing in oven at 105°C overnight.

6.6 **Volumetric flasks**: of various sizes with ground glass stoppers.

6.7 **Vials**: 40 ml, with pierceable Teflon screw cap top.

6.8 **Spatulas**: Stainless steel.

6.9 **Disposable pipettes**: Pasteur.

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6.10 **Magnetic Stir Bars:** VWR brand cat no. 58948-116

6.11 **Purge and trap devices**: (A) Tekmar 3100 Purge and Trap Concentrator. (B) Archon Purge and Trap Auto-Sampler System (Varian)

6.11.1 The Archon Purge and Trap Auto-sampler is designed to automate the tedious sample handling procedures associated with purge and trap analyses for volatile organic compounds (VOCs) under current EPA methods. The Archon can be used for drinking water, wastewater, soil and solid analyses.

Function Keys: A) **Method**: Methods are entered using a keypad with LCD Screen. All information can be edited using keypad. Settings are variable dependent on analysis requirements

Method parameters consist of:

	MS1/MS3/MS4	MS1/MS3/MS4
Sample Type (water/soil)	soil (Low level)	water/soil (High level)
First Vial (1-51)	variable	variable
Last Vial (1-51)	variable	variable
Sample Volume (0-25ml)	05	05
Dilution Factor	variable	variable
Rinse Volume (0-25ml)	--	07
# Rinses (0-20)	--	01
Standard 1(yes/no)	variable	variable
Standard 2(yes/no)	variable	variable
S. Preheat Stir(yes/no)	no	no
Stir(yes/no)	yes	no
W. Stir Time(0-9.9min)	0.0	0.0
W. Settle Time(0-99sec)	0.0	0.0
Syringe Flush(0-20)	00	01
Preheat(yes/no)	yes	no
Preheat Temp(amb-140C)	40	--
Preheat Time(0-99.9min)	1.0	--
Purge Time(0-999.9min)	11	--
Desorb Time(0-99.9min)	1.0	1.0
Operation Mode(Local/Remote)	Remote	Remote
Cycle Timer(0-99.9min)	0.0	0.0
Aux Timer(0-999min)	0.0	0.0
Link to Method	variable	variable

B) **Auto**: Starts purge sequence. Initiates single blank runs.
Single blank runs can be programmed after specific sample vials within a particular method. These method blanks allow the cleaning of not only the Archon, but also the purge and trap and GC. The blank water is withdrawn from the blank water reservoir, which is pressurized by helium gas at 20-25PSI. Before changing the blank reservoir, the "Blank Water Reservoir and Standard Helium" switch must be turned off. This switch is located on the right rear panel inside the Archon. After refilling the reservoir this switch must be turned on.

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C) **Manual**: allows for manual injection of samples.

D) **Flush**: allows flushing of Archon components using water from Blank Water Reservoir.

E) **System**: provides access to all settings, maintenance, calibration, and options of the Archon.

F) **Pause/Stop**: allows analyst to pause or abort sequence.

6.11.2 The sample is purged in a 40 ml VOA vial by the Archon for the Low Level analysis. The sample is purged in a 5 ml sparge tube for the high level analysis. The purge gas passes through the water column as finely divided bubbles, each with a diameter of less than 3 mm at the origin. The purge gas is introduced no more than 5 mm from the base of the water column.

6.11.3 The trap is at least 25 cm long and has an inside diameter of at least 0.105". This trap is commercially available from Supelco (Vocarb 3000, Catalogue No. 2-1066 for system A and No. 2-4920 for system B).

6.11.4 The desorber is capable of rapidly heating the trap to 225°C and performing the bake out step at 260°C.

6.11.5 The chamber heater is capable of maintaining the purge device at 40°C ±1°C.

6.12 **Gas chromatograph**: HP5890 Series II or HP-6890. A complete analytical system with a temperature programmable gas chromatograph suitable for on-column injection and all required accessories including syringes, analytical columns and gases.

6.13 **Column**: (A). 75 M x 0.53 mm ID or (B). 30M x 0.25 mm ID, DB624 commercially available from J&W Scientific (or equivalent).

6.14 **Mass spectrometer**: HP5971MSD or HP5972MSD- Capable of scanning from 35 to 260 AMU every 1 second or less, utilizing 70 volts electron energy in the electron impact ionization mode and producing a mass spectrum which meets all the criteria listed in section 11.1 when 50 ng of 4-Bromofluorobenzene (BFB) is injected through the gas chromatograph inlet.

6.15 **Data system**:

6.15.1 **Computers:** The Volatiles laboratory has three GC/MS systems analyzing method 5035/8260B. VOA MS1 has an AST computer with a Windows 95 operating system. VOA MS3 and MS4 have DELL computers with Windows NT operating system. All computer systems are networked to a Windows 2000 server, which is the destination of all files. A differential back-up is performed nightly and a full back is performed each weekend using Veritas Backup Exec to tapes. As the systems acquires and stores data onto the server, the server becomes full. The data is downloaded and archived onto CDs.

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6.15.2 **Software:** HP Enviroquant MSChemstation - The software is interfaced to the mass spectrometer detector and allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer software allows searching of any GC/MS data file for ions of a specified mass and plotting such ion abundance versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). The software is also capable of integrating the abundance in any EICP between specified time or scan number limits. The most current version of the EPA/NIST Mass Spectral Library is also available. Current versions VOA MS1:G1032C version C.01.00, VOA MS3:G1701BA Version B.01.00, and VOA MS4: G1701AA version A.03.00.

7.0 REAGENTS AND STANDARDS

7.1 Reagents:

7.1.1 **Methanol**: Purge and Trap grade, purchased from Fisher, Catalog No.A453-500 or equivalent.

7.1.2 **Reagent Water**: Water in which interference is not observed at the method detection limits (MDL) of the parameters of interest. Tap water, which has been passed through activated carbon (12-20) mesh, meets the above criteria. Note: activated carbon purchased from Aldrich (cat. # 24,224-1).

7.2 Standards:

7.2.1 **Primary Standards**: obtained from a commercial source. Store in the freezer at -10°C to -20°C. After opening; store in a vial with a mininert valve. All Certificates of Analysis (COAs) are to be marked with the appropriate Primary Standard ID and recorded in the primary standard logbook. Primary standards are not to be used after the manufacturer's expiration date. Once a standard has been opened, it may not be used after 6 months. Gas mixtures are to be regularly monitored for degradation. A copy of the (COAs), which details the compounds found in the mixes, is attached.

Manufacturer	Standard	Catalog Number	Conc. (µg/ml)
Restek	Ketones	30006	5000
Restek	Acrolein	30645	5000
Restek	Oxygenates	30465	2000/10,000 (TBA)
Restek	Custom	558360	2000
Restek	Gases	30042-510	2000
Restek	Cal Mix#1	30633	2000
Restek	2-CEVE	30265	2000
Restek	Vinyl Acetate	30216	2000
Supelco	1,4-Dioxane	44-2251	10,000*
Ultra	Surrogate	STM-530-I	2500
Ultra	1,3,5-Trichlorobenzene	AS-E0176	5000

*The primary standard for 1,4-Dioxane is made up from neat by diluting 10 µl of neat 1,4-Dioxane into a 1.0 ml volumetric flask.

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7.2.2 **Working Calibration Standard**: The working calibration standard is prepared and stored in micro reaction vessels or vials capped with mininert valves. It is also checked frequently for signs of degradation and evaporation. Working calibration standards expire after 6 months. This standard is used for preparing calibration curves and daily continuing calibrations.

7.2.2.1 **High Level and Low Level Method**: Into a 5 ml volumetric flask, add the following: 125 µL of Ketone standard and 62.5 µL each of gas, Custom standard, Cal Mix #1, Vinyl acetate, and Oxygenate standards, 312.5 µL of 2-CEVE standard, 25µL of Acrolein std, 25µL of 1,3,5-Trichlorobenzene, 237.5 µL 1,4-Dioxane and 50 µL of surrogate standard. Volumize to 5ml with MeOH. Final concentration equals 125 µg/ml for the Ketones and 2-CEVE, 500 µg/ml of 1,4-Dioxane, and 25 µg/ml for all other analytes including surrogates.

7.2.3 **Secondary Source/BS/Matrix Spike Standards**: obtained from a commercial source. They are stored in the freezer at -10°C to -20°C. After opening; they are stored in a vial with a mininert valve.

Manufacturer	Standard	Catalog Number	Conc. (µg/ml)
Accustandard	TCL Ketone Mix	CLP-022K-25x	5000
Ultra	Acrolein	RCC-150	10,000*
Accustandard	Method 502.2 VOC	M-502-10x	2000
Ultra	VOC Mix	DWm-592-1	2000
Ultra	Custom Std	CUS-10604	2000
Accustandard	2-CEVE	M-601C-10x	2000
Supelco	Vinyl acetate	4-0327	5000
Ultra	1,4-Dioxane	RCC-180	10,000*
Accustandard	tert-Butyl Alcohol	S-410	2000

* The primary standard for 1,4-Dioxane is made up by diluting 10 µL of neat 1,4-Dioxane into a 1.0 ml volumetric flask.

7.2.3.1 **Initial Calibration Verification Standard (ICV)/Blank Spike (BS)/Matrix Spike (MS) Standard (High Level Method):** Used to check the primary standard. Into 5ml volumetric flask containing approximately 3 ml of MeOH add 62.5 µl each of Custom standard, VOC mix, 502.2 Mix and Vinyl acetate; 125 µL of Acrolein, and 250 µL of 1,4-Dioxane. Add 100 µL Ketone Mix. Add 312.5 µL tert-Butyl Alcohol and 2-CEVE, Volumize to 5ml with MeOH. Individual components will have a final concentration of 25 µg/ml with the exception of 1,4-Dioxane at 500µg/ml, 2-CEVE and Ketones at 125 µg/ml. A copy of the COAs, which details the compounds in the mixes, is attached.

7.2.4 **Surrogate Standards**: The surrogate solution contains Dibromofluoromethane, Toluene-d_8, 4- Bromofluorobenzene, and 1,2-Dichloroethane-d_4. The mixed stock surrogate solution is purchased from Ultra at 2500 µg/ml (cat. no. STM-530-1). The stock solution is logged into the primary standard logbook upon receipt. Two surrogate standard solutions are prepared from the stock at different concentrations in methanol:

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7.2.4.1 **Low level analysis- Surrogate/Internal standard QC mix**: This solution is prepared by adding 250 µL from the stock surrogate (7.2.4) and stock internal standard (7.2.5) to a 5 ml volumetric flask containing methanol and diluting to give a final concentration of 125 µg/ml. Transfer this standard to reservoir #1 on the Archon. For the low-level method, addition of 1 µL of this standard by the Archon auto-sampler to 5.0 ml of sample is equivalent to 25 ppb.

7.2.4.2 **High level analysis-** surrogates from the primary standard are added directly to the methanol extract. 15 µL of the primary surrogate standard is spiked into 15 ml of methanol. (30 µL is used if the sample vial contains 30 ml of methanol.

7.2.5 **Internal Standards**: The internal standard solution is purchased from Restek at 2,500 µg/ml (cat no. 30074) and contains Pentafluorobenzene, Chlorobenzene-d_5, and 1,4-Dichlorobenzene-d_4. Two internal standard solutions are prepared from the stock at different concentrations in methanol.

7.2.5.1 **Internal Standard QC mix**: This solution is prepared by adding 250 µL from the stock Internal Standard to a 5 ml volumetric flask containing methanol. When brought to volume with methanol the final concentration is 125 µg/ml. Transfer the solution to reservoir 1a on the Archon. Addition of 1µL of this standard by the Archon auto-sampler to 5.0 ml sample is equivalent to 25ppb.

7.2.5.2 **Internal Standard for calibration:**

7.2.5.2.1 **Low Level Method**: Add 50µL of the stock solution (7.2.5) to the 5ml of methanol in a 5 ml volumetric flask to give a concentration of 25µg/ml of each internal standard compound. Addition of 10µL of this standard to 10.0 ml of organic free water is equivalent to 25ppb.

7.2.5.2.2 **High Level Method:** 1µL of internal standard QC mix (7.2.5.1.1) is injected by the Archon into all calibration standards for a final concentration of 25 ppb.

7.2.6 **4-Bromofluorobenzene (BFB) Standard**: A stock solution in methanol is purchased from Supelco at a concentration of 2,000 µg/ml (cat. no. 4-8083).

7.2.6.1 Add 62.5µL of the stock solution to a 5.0-ml volumetric flask and dilute with methanol to produce a 25-µg/ml solution. The tuning solution is purged by the Archon by adding 10µL to 10ml organic free water for a final concentration of 25 µg/L.

7.2.7 **Matrix Spiking Standard**: prepared as in section 7.2.3.1.

7.2.7.1 **Low Level MS/MSD,** 10 µl of the matrix spike solution added to the vial. Final spike concentration is 25 µg/L.

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7.2.7.2 **High Level MS/MSD**, 100 µl of the matrix spike solution is added to 100 ml of DI water containing 1 ml of sample extract. Transfer to two 40 ml vials for the MS and MSD. Final spike concentration is 25 µg/L. See attachment H for MCP requested MS/MSD.

8.0 PROCEDURE

8.1 Initial calibration:

8.1.1 ESS Laboratory's policy is that the audit trail on the Chemstation/Enviroquant software is always on. This ensures that any changes made to the instrument operating method be documented through the audit trail.

8.1.2 GC/MS operating conditions: The following parameters are setup through Hewlett Packard MS EnviroQuant. A copy of an EnviroQuant method is attached.

Electron energy:	70 volts		
Mass range:	35 amu - 260 amu		
Scan time:	1.5 sec/scan		
Initial column temp..:	35°C - 37°C		
Initial column hold time:	1.2 min.		
Column temp. program:	Rate	Final Temp	Final Time
Level 1	9°C/Min	150°C	0 min
Level 2	35°C/Min	220°C	2 min
Injector temperature:	200°C		
Transfer line temperature:	280°C		
Carrier gas (Helium):	15 cm^3/ml		

8.1.3 Assemble a purge-and-trap device that meets the specification in Section 6.11. New traps are conditioned one hour at 270°C in the bake mode. Prior to daily use bake trap for 8 minutes at 260°C.

8.1.4 Just prior to any analysis of samples or standards, the run logbook should be completely filled out (See Attachment A). Each instrument has its own run logbook. Any unusual observations are included in the comment section of the logbook. The information that must be recorded is:

8.1.4.1 **Batch Date**: Date analytical sequence set-up

8.1.4.2 **Vial #**: Position the standard/sample vial is placed in the auto-sampler

8.1.4.3 **File #**: File ID assigned to standard/sample in sequence

8.1.4.4 **Lab ID**: The ID of the sample or standard. Sample IDs are assigned at log-in by ELEMENT (ex: 0508001) and Batch QC samples contain the batch ID (ex BG50801-blk1, BG50801-bs1, BG50801-bsd1, ...)

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8.1.4.5 **Method**: Record the method used. The method includes instrument operating parameters and initial calibration for quantitation.

8.1.4.6 **Comments/Dilution/Standard ID**: Record comments and unusual observations. Record any dilution information in the comments section. (Ex. 25x dilution on aqueous sample: 0.2/5 ml) Record standard/spike ID for Tune, CCV, BS/BSD, and MS/MSD.

8.1.4.7 **Analyst**: Analyst must record their initials.

8.1.5 A tune check must be performed before the initial calibration curve. This is accomplished by purging a 5 µg/L standard of the 4-Bromofluorobenzene standard (See section 7.2.6). Analysis is not to begin until the 4-BFB spectra meets the criteria in Section 11.1. Evaluation of the ion abundance is performed by using one of the following scan scenarios:

- Use one scan at the peak apex,
- Use the one scan either directly preceding or following the apex,
- Use the mean of the apex and the scans preceding and following the scan, or
- Use the average across the entire peak.

8.1.5.1 The analysis of all calibration standards must be accomplished within 12 hours of this tune check.

8.1.6 Prepare the standards for generating the calibration curve using the Working Standards (see section 7.2.2) and Internal Standard solution (see Section 7.2.5) as follows:

8.1.6.1 **For the Low Level Soil Method:** Initial calibration standards are prepared in VOA vials containing a magnetic stir bar. Seven standards are analyzed at concentrations of 1.0, 2.0, 5.0, 10, 25, 50, and 100 ppb. The 1.0, 2.0, 5, 10, 25, 50, and 100 standards are prepared by adding 0.4, 0.8, 2.0, 4.0, 10.0, 20.0 and 40.0µL of working calibration standard (§ 7.2.2.2) to 10ml of water. 10µL of internal standard is added to each standard for a final concentration of 25ppb. The vials are capped, heated to 40°C and purged by the Archon auto-sampler. All calibration standards are stirred by the Archon.

8.1.6.2 **High Level Calibration Curve**: Initial calibration standards are prepared in 50 ml volumetric flasks containing 500µL MeOH. Seven standards are analyzed at concentrations of 0.4, 0.5, 2.0, 10, 25, 50, and 100ppb. The 0.4 ppb standard is prepared by adding 0.8 µL of 25 ppm working calibration standard to 50ml water. The 2.0, 10, 25, 50, and 100-ppb standards are made by adding 4, 20, 50, 100, and 200µL of 25 ppm working calibration standard (§ 7.2.2.2) to 50ml of water. The 0.5 ppb standard is prepared by adding 1 µL of 25 ppm working standard to 50 ml of DI water. After bringing calibration standards to volume with DI water, invert 3 times, and transfer to 40 ml VOA vials. The standards are purged according to method 5030B.

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NOTE: The Archon auto-sampler injects 1 µL of internal stand QC mix (7.2.5.1.1) into each calibration standard (5.0 ml) before purging for a final concentration of 25 ppb.

8.1.7 Carry out the purge-and-trap analysis procedure as described in Section 8.3.

8.1.8 EnviroQuant will tabulate the area response of the characteristic ions (see Table 2) against concentration for each compound and internal standard. It will also calculate response factors (RF) for each compound relative to its internal standards according to the calculations in Section 9.1.

NOTE: EnviroQuant software calculates and reports response factors for the analyst. It is each analyst's responsibility to become familiar with the software by reading the available manuals. **After generating the initial calibration curve in Enviroquant, the analyst must visually check that each calibration standard was entered into the new calibration method. This is accomplished by checking that the area response for one compound from each calibration standard's printout corresponds to the area count listed in the calibration method in Enviroquant.** All of the analytes along with their retention times and the ions used for qualitative analysis are listed in attached method.

8.1.9 The average RF is calculated in the EnviroQuant Method for each compound. A system performance check is made before this calibration curve is used. Five compounds (System Performance Check Compounds or SPCCs) are checked for a minimum average response factor. The minimum acceptable RFs are 0.1 for Chloromethane, 1,1-Dichloroethane, and Bromoform, and 0.3 for Chlorobenzene and 1,1,2,2-Tetrachloroethane. These compounds are used to check compound instability and to check for degradation of the system, such as active sites in transfer lines or trap failure. ***NOTE: The minimum response factor for each compound should not be less than 0.01. ESS Laboratory has found that the following poor performing compounds do not meet this criterion: Acetone, 2-Butanone (MEK), and 1,4-Dioxane.***

8.1.10 Using the RFs from the initial calibration, EnviroQuant software calculates the percent relative standard deviation (%RSD) for all compounds using the equation in Section 9.2. The %RSD should be less than 15% for each compound, except for the following continuing calibration compounds (CCCs) 1,1-Dichloroethene, Chloroform, 1,2-Dichloropropane, Toluene, Ethylbenzene and Vinyl Chloride, where the %RSD should be less than 30%

8.1.10.1 Linearity - If the %RSD of any compound is 15% or less, then the relative response factor is assumed to be constant over the calibration range, and the average relative response factor may be used for quantitation.

8.1.10.2 When the RSD exceeds 15%, the plot and/or visual inspection of the calibration data can be a useful diagnostic tool. The inspection may indicate analytical problems, including errors in standard

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preparation, the presence of active sites in the chromatographic system, analytes that exhibit poor chromatographic behavior, etc.

8.1.10.3 If a %RSD greater than 30 percent is measured for any method analyte (50% for poor purgers), then corrective action to eliminate a system leak and/or column reactive sites is required before re-attempting calibration. See section 1 for poor purgers.

8.1.10.4 If the RSD of ANY compound exceeds 15% (never to exceed 30% except for poor purgers), then one of the following options must be applied to the GC/MS initial calibration in this situation or a new initial calibration must be performed.

8.1.10.4.1 Adjust the instrument and/or perform instrument maintenance until the RSD of the calibration meets the 15% QC limit. This option would apply in those instances where a linear instrument response is expected.

8.1.10.4.2 Narrow the calibration range until the response is linear. If the low standard is below the estimated quantitation limit (i.e., for the poor purgers in a commercially available prepared standard mix), then this standard may be dropped. Recalculate the RSD without this standard to see if the RSD meets the QC limit.

8.1.10.4.3 Use a linear calibration. This would be achieved by performing a linear regression of the instrument response versus the concentration of the standards, **do not** force the curve through zero. In order to be used for quantitative purposes, the Correlation of Determination (COD/R^2) must be greater than or equal to 0.99.

8.1.10.4.4 Use a calibration curve or non-linear calibration model. This option should be used only after exhausting the other three options, or in situations where the analyst knows that the instrument response does not follow a linear model over a sufficiently wide working range. Correlation of Determination (COD/R^2) must be greater than or equal to 0.99. ***When non-linear calibration is employed a minimum of six calibration points must be used for second order and seven calibration points for third order.***

8.1.11 Immediately after generating the initial calibration, a second source calibration verification standard (§ 7.2.3.1) must be analyzed to confirm the initial calibration. The percent difference for the CCC analytes must be within 20% (30% for all other target analytes). ***When samples are to be run for the DoD, all compounds must have ≤25% difference with no allowance.***

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8.1.12 The RRT's are established from the 25 ppb mid-point standard in the initial calibration

8.2 **Daily GC/MS analysis**: Just prior to all samples and standard analysis, the run logs are filled out according to 8.1.4.

8.2.1 Prior to the analysis of samples, purge a 25 µg/L standard of the 4-Bromofluorobenzene (Section 7.2.6). The resultant mass spectra for BFB must meet all of the criteria in Section 11.1 before sample analysis begins. This criteria must be met each 12-hour shift.

8.2.2 The initial calibration curve must be verified each 12-hour shift. This is accomplished by analyzing a continuing calibration verification standard (CCV) at **25 µg/L for both high and low level soil analyses**. Determine that the SPCC meet criteria from Section 8.1.9 and the CCCs listed in Section 8.1.10 have %Drift/Deviation less than 20% with all other analytes having a % Drift/Deviation less than 30%. Compounds not analyzed for in the batch of samples do not need to meet the criteria. Continuing calibrations used for 8021 analysis must have <20% Drift/Deviation. ***When samples are to be to be run for the DoD, all compounds and surrogates should have <20%*** Drift/Deviation***, see section 11.4 for expanded criteria.*** For compounds analyzed for 624 continuing calibration criteria are met or exceeded by 8260 criteria. **Refer to Attachment K for Mass. DEP MCP requirements.**

8.2.3 The internal standard responses and retention times in the continuing calibration standard (CCV) should be evaluated immediately after data acquisition. If the area for any of the internal standards changes by a factor of two (-50% to +100%) or the retention time changes by ±0.5 minutes from the **mid-point standard of the initial calibration**, the mass spectrometer should be checked for defects. The relative retention time of each compound in each calibration run must agree to within 0.06 Relative Retention Time (RRT) Units. The RRT's in the continuing calibration standard is compared to the RRT's from the 25 ppb standard in the ICAL. Analysis is not to continue until problem has been resolved.

8.2.4 Method blank analysis should be performed at the following frequency and within these criteria:

8.2.4.1 For the analysis of volatile compounds, a method blank analysis must be performed once for each 12-hour shift and must be analyzed before any samples.

8.2.4.2 A method blank for volatile analysis should contain less than or equal to the Method Reporting limit of methylene chloride (see Att. K for Mass DEP). For all other TCL compounds, the method blank should contain less than ½ MRL of any single TCL analyte. See section 11.5 for instructions on corrective action when criteria are exceeded.

8.2.4.3 Blank Spike/Blank spike duplicate analysis must be performed once for each 12-hour shift, after the tune and CCV, and must be analyzed

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before any samples. BS/BSD concentrations are to be at the midpoint (median standard level) of the calibration curve See section 10.0 for BS/BSD acceptance criteria.

8.2.4.3.1 **Low Level BS/BSD,** 10 µl of the BS/BSD solution (§ 7.2.3.1) added to each of two VOA vials containing 5 ml of reagent water. Final spike concentration is 25 µg/L.

8.2.4.3.2 **High Level BS/BSD**, 100 µl of the BS/BSD solution (§ 7.2.3.1) is added to 100 ml of DI water containing 1 ml of MeOH. Transfer to two 40 ml vials for the BS and BSD. Final spike concentration is 25 µg/L.

8.3 **GC/MS analysis**: All samples and standards are analyzed under the same conditions in the associated initial calibration. Run logs are filled according to section 8.1.4.

8.3.1 Soil/Sediment Samples

8.3.1.1 All samples and standard solutions should be allowed to warm to ambient temperature before analysis.

8.3.1.2 Set up the GC/MS system as outlined in Section 8.1.2.

8.3.1.3 BFB tuning criteria and daily GC/MS calibration criteria must be met before analyzing samples.

8.3.1.4 Sample loading

8.3.1.4.1 **Low Level Analysis**: Sample vials containing approximately 5g soil, 5ml water and 1 magnetic stir bar are loaded directly onto the Archon. The Archon is programmed to stir the samples.

8.3.1.4.2 **High Level Analysis**: Samples must have been spiked with surrogate and, when applicable, matrix spike solution. The analyst will take a 500 µL aliquot of methanol extract directly through the septa of the sample vial. Transfer the aliquot into a 50 ml volumetric flask containing DI water. Volumize with DI water, invert 3 times, and transfer into a clean 40m VOA vial. The vials are loaded into the Archon for analysis by method 5030B

8.3.1.5 Internal Standards

8.3.1.6 Low Level Method: The Archon is programmed to add 1 µL Surrogate/Internal standard (§ 7.2.4.1.1) to the sample. The Archon also adds an additional 5 ml DI water to each vial.

8.3.1.7 High Level Method: Five (5) ml of the sample dilution (§ 8.3.1.4.2) is transferred by the Archon to the 5 ml sparge tube.

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The Archon is programmed to add 1 µL internal standard (§ 7.2.5.1.1) to all standards, blanks, and samples.

8.3.1.8 Purge the sample for 11.0 ± 0.1 min at ambient temperature. At the conclusion of the purge time, the purge-and-trap will sound a ready signal from the GC and begin to desorb the trap while initiating the chromatographic temperature program and the CHEMSTATION data acquisition.

8.3.1.9 After the sample has been desorbed for 1 minute, the 3100 will recondition the trap by heating to 260°C while back flushing with Helium. This step will take 10 minutes.

8.3.1.10 For the high level method if any sample analyte concentration exceeds the initial calibration range, the sample must be re-analyzed at a higher dilution. If the low-level sample produces results above the calibration range the methanol extract must be analyzed. To dilute a methanol extract, a smaller amount of methanol can be taken from the sample vial and re-analyzed. Record dilutions in the Run logbook. All dilutions should keep the response of the major constituents in the upper half of the linear range of the curve.

8.3.1.11 When a sample is analyzed that has any target compound at a level exceeding twice the initial calibration range, this analysis should be followed by a blank reagent water analysis to demonstrate that system is clean. If the blank analysis is not free of interferences, the system should be decontaminated. Sample analysis may not resume until a blank can be analyzed that is free of interferences. Analysis of sample in the contaminated chamber may not resume until the analysis of reagent water in the chamber is free of any target compounds.

NOTE: Since the analytical system is automated the analyst may not be aware that a high concentration sample has been analyzed. If analysis of subsequent samples occurs, then the presence of the high concentration analyte makes reanalysis of that sample necessary to determine if carryover has occurred.

8.3.1.12 **For matrix spike analysis see section 7.2.7.**

8.4 Data interpretation

8.4.1 Qualitative identification

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8.4.1.1 An analyte (Table 2) is identified by comparison of the sample mass spectrum with a standard's mass spectrum obtained on the user's GC/MS. Reference spectra are obtained through analysis of the calibration standards. To compare the sample and reference spectra, go into the EnviroQuant software (QEDIT). The characteristic ions from the reference spectra are defined to be the three ions of greatest

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relative intensity - any ions over 30% if less than three such ions are present.

8.4.1.2 The requirements for qualitative verification by comparison of mass spectra are:

8.4.1.2.1 The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.

8.4.1.2.2 The RRT of the sample component is compared to the RRT from the 25 ppb standard in the ICAL and must be within ±0.06 RRT units of the RRT of the standard component.

8.4.1.2.3 The relative intensities of the characteristic ions agree to within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%).

8.4.1.2.4 Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs. ESS Laboratory currently reports para and meta Xylenes due to inability of resolving the isomers.

8.4.1.2.5 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When chromatographic peaks obviously represent more than one component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra are important. Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra, and in qualitative identification of compounds. When analytes co-elute (i.e., only one chromatographic peak is apparent), the identification criteria can be met, but each analyte spectrum will contain extraneous ions contributed by the co-eluting compound.

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8.4.1.2.6 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the type of analyses being conducted. When serving the role of QA (or referee) laboratory, tentatively identified compounds (TICs) shall always be reported. Guidelines for making tentative identification are:

- Relative intensities of major ions in the reference spectrum (ions >10% of the most abundant ion) should be present in the sample spectrum.
- The relative intensities of the major ions should agree within ±30% to be consistent with target compound list identification. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 20 and 80%).
- Molecular ions present in the reference spectrum should be present in the sample spectrum.
- Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.
- Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

8.4.2 Quantitative Analysis

8.4.2.1 Identified compounds are quantified by the internal standard method, utilizing the integrated abundance of the primary ion. The internal standards are listed in Section 7.2.5. The EnviroQuant method is setup to calculate the concentration of the analytes of interest in ng/ml using the initial calibration results stored in the method (see attached method). This raw data number does not account for deviations in sample volume, sample weight, dilution factors, and % moisture. To include these other factors, see section 9.0.

9.0 CALCULATIONS

9.1 Response Factor (RF)

RF= (As x Cis)/(Ais x Cs)

Where:

A_S = Peak area (or height) of the analyte or surrogate.

A_{IS} = Peak area (or height) of the internal standard.

C_S = Concentration of the analyte or surrogate, in µg/L.

C_{IS} = Concentration of the internal standard, in µg/L.

9.2 Percent Relative Standard Deviation (%RSD)

$$\% \text{ RSD} = (\text{SD}/ \text{RF}_{\text{Average}}) \times 100\%$$

Where:

RSD = Relative standard deviation.
RF = Mean of 5 initial RFs for a compound.
SD = Standard deviation of average RFs for a compound.

9.3 Percent Difference

$$\%\text{Difference} = ((\text{RF}_{\text{Average}} - \text{RFc})/ \text{RF Average}) \times 100\%$$

Where:

$RF_{Average}$ = Average response factor from initial calibration
RF_c = Response factor from current verification check standard.

9.4 Percent Drift

$$\%\text{Drift} = ((C1-C0)/ C1) \times 100\%$$

Where:

C_1 = compound standard concentration
C_0 = measured concentration using selected quantitation method

9.5 Concentration of Target Analytes in Water and Water-Miscible Waste Concentration

$$\mu g/L = (As)(Cis)(D) / (Ais)(RF_{Average})(Vs)(1000)$$

Where:

A_S = Area (or height) of the peak for the analyte in the sample.

A_{IS} = Area (or height) of the peak for the internal standard.

Cis = Concentration of the internal standard in the concentrated sample extract or volume purged in µg/L.

D = Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution was made, D = 1. The dilution factor is always unit less.

$RF_{Average}$ = Mean response factor from the initial calibration.

V_S = Volume of the aqueous sample extracted or purged (ml). If units of liters are used for this term, multiple the results by 1000.

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Using the units specified here for these terms will result in a concentration in units of ng/ml, which is equivalent to µg/L.

9.6 Concentration of Target Analytes in Sediment/Soil, Sludge, and Waste

9.6.1 High-concentration procedure concentration

$$\mu g/Kg \text{ dry wt} = \frac{(As)(Cis)(Vt) / (Ais)(RF_{Average})(Vi)(Ws)}{(\%S/100)}$$

Where:

A_S, Ci_S, A_{IS}, $\overline{RF}$ are the same as in water and water-miscible waste above.

V_t = see section 2.7.

V_i = Volume of extract added (500 µL or lower when a dilution is need) for purging.

W_S = Weight of sample extracted or purged (g). The wet weight or dry weight may be used, depending upon the specific applications of the data.

%S = % Solids

9.6.2 Low-concentration procedure concentration

$$\mu g/Kg \text{ dry wt} = \frac{(As)(Cis)(Vt) / (Ais)(RF_{Average})(Vi)(Ws)}{(\%S/100)}$$

Where:

A_S, C_{IS}, A_{IS}, RF_{Ave}, W_S are the same as in water and water-miscible waste above.

V_t = Volume of total extract (µL) (use 10,000 µL for volume of DI purged)

9.7 Percent Solids

%dry weight = ((g of dry sample)/ (g of sample)) x 100%

9.8 Percent moisture

% Moisture = 100 - % Solids

10.0 QUALITY ASSURANCE/QUALITY CONTROL

Accuracy and Precision

All laboratory personnel must demonstrate initial proficiency for each sample preparation method/matrix that he or she performs. All new employees must successfully demonstrate initial proficiency prior to independently performing analysis on real samples. This must be accomplished by generating data of acceptable accuracy and precision for target analytes in

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a clean matrix. The initial proficiency results will become part of each employee's training file.

QC Sample Preparation:
Spiking Solution: Four QC samples must be prepared from a spiking solution with the analytes of interest. The spiking solution must be made using standards **prepared independently from those used for calibration**. The samples must be prepared at a concentration that world result in data falling within the middle of the calibration curve.

Sample Analysis:
The four QC samples must be analyzed within the criteria of the method being evaluated. The QC samples must be handled in exactly the same manner as actual samples.

Accuracy Calculation:
Accuracy is defined as the closeness of agreement between an observed value and an accepted reference value. Each of the four spiked samples will be calculated for percent recovery. The average of the percent recovery values is the accuracy result.

Precision Calculation:
Precision is defined as the agreement of a set of replicate measurements without assumption of knowledge of the true value. Precision is estimated by the relative standard deviation (RSD) of the four QC samples.

$$\%RSD = (s / \bar{x})\ 100\ \%$$

Where:

s = Standard Deviation of a finite number of values. On a scientific calculator use the **σxn-1** key.

$\bar{x}$ = the average of the four QC sample % recoveries.

Reporting Accuracy and Precision
Accuracy and Precision data should be presented with the following minimum info:

Matrix: Prep Method:
Analysis Method: Date Analyzed:

Parameter	*% Rec. QC 1*	*% Rec. QC 2*	*% Rec. QC 3*	*% Rec. QC 4*	*Average Recovery*	*Standard Deviation*	*%RSD*

Interpretation of Results: Percent recovery is to be within 75-125% for all analytes with a RPD of ≤20%. For the full 5035/8260B list a maximum of 5 sporadic marginal failures are allowed with the expanded criteria of 60-140%.

10.1 A preparation batch of samples is defined as a group of up to twenty field samples of similar matrix type that have been prepared at the same time or time sequence with the same lots of reagents for the same analysis. In addition to the twenty samples, each preparatory batch will contain at a minimum, a method blank, a blank spike, a

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matrix spike, and a matrix spike duplicate. An analytical or instrumental batch is defined as samples that are analyzed together within the same time period or in continuous sequential time periods. Within the analytical batch are included individual QC requirements as defined by the analytical (determinative) method. For instance, each injection sequence would begin with a tune & CCV (or initial 6-point calibration and ICV), followed by a method blank, the other QC samples (normally a BS, MS, MSD, etc.), and the remaining field samples. Preparation batches of samples may be continuously strung together in the run sequences, as long as the analytical batch QC requirements meet the acceptance criteria established within this SOP. If all field and QC samples can not be completed within the 12-hour clock, then another tune, CCV, and MB are required. Each analytical sequence must be documented using the run log in Figure 1.

10.2 Perform BFB tune every 12 hours. Tuning acceptance criteria are presented in Section 11.1. The computer software will evaluate the tune information. The analyst should be aware of the process used. The criteria specified in Section 11.1 would typically be derived using three scans, one at the apex, and one on both sides of the apex.

10.3 Run a 7-point initial calibration curve, using the primary source standards each time major instrument maintenance occurs, or if the CCV does not meet acceptance criteria. Acceptance criteria are presented in Section 11.2.

10.4 Run initial calibration verification (ICV) standard using secondary standards (7.2.3) immediately following the initial calibration curve. Acceptance criteria are listed in Section 11.3.

10.5 Run a mid-point Continuing Calibration Verification (CCV) using the primary source standards on a daily basis after the BFB tune and before sample analysis. Also run a CCV every 12 hours during an analytical sequence. Acceptance criteria are listed in Section 11.4.

10.6 A method blank must be prepared with each batch of samples not to exceed 12 hours. The method blank should be prepared from organic-free water and all reagents in the same ratios used to prepare samples. The method blanks are to be related to each 12-hour sequence of samples injected. Acceptance criteria for these blanks are listed in Section 8.2.4 and corrective action is listed in Section 11.5. Instrument blanks may be injected at any time in the sequence to verify absence of contamination.

10.7 A Blank spike (BS) and Blank spike duplicate (BSD) must be prepared and analyzed with each batch of samples. The BS/BSD are prepared using a source different than that used to prepare the initial calibration standards and contain all method target analytes. Acceptance criterion is 70-130% and ≤25% RPD (≤20 for Mass DEP MCP). ***For Mass DEP MCP, all compounds in the BS/BSD must be between 70-130%Rec with allowances for difficult analytes (see Attachment K). Sporadic marginal exceedances have the expanded criterion of 40-160%. See Table 3 for DoD blank spike acceptance criteria. Sporadic Marginal Exceedance number is as follows:***

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Number of Analytes in BS	Allowable number of Marginal Exceedances
>90	5
71-90	4
51-70	3
31-50	2
11-30	1
<11	0

Exceedances should be within marginal exceedance limits (Table 3) or expanded criterion listed above.

10.7.1 Control charts will be maintained for the BS/BSD for a representative subset of target analytes and surrogate spikes. Biannually, control limits will be determined for all target analytes and surrogates for comparison to default limits.

10.8 On an ongoing basis the laboratory analyzes matrix spikes and matrix spike duplicates from each batch of 20 samples.

10.8.1 The matrix spike is prepared as described in Section 7.2.7. ***NOTE: MA MCP requires a special preparation when the client requests that MS/MSDs are performed on their project. See Attachment H for this procedure.***

10.8.2 Matrix spike control limits are 70-130%. %RPD between spike and spike duplicate should be ≤30%. ***For DoD matrix spike acceptance criteria use the blank spike acceptance criteria in Table 3.*** For samples outside control limits, see Section 11.6 for corrective action.

10.9 Surrogates recoveries limits are 70-130%. ***For DoD surrogate acceptance criteria use the blank spike acceptance criteria in Table 3.*** For samples outside control limits, see Section 11.7 for corrective action.

10.10 The relative retention times (RRTs) need to be checked for each identified compound in samples, and compared to standard RRT. Acceptance criteria are presented in Section 11.8.

10.11 Ion abundance for target compounds and any tentatively identified compounds need to meet specific requirements. Acceptance criteria for ion abundance are presented in Section 11.9.

10.12 Internal standard area counts for standards and samples must meet specifications as described in Section 11.10.

10.13 Data shall be checked to ascertain if it conforms to accepted practices. All sample analytical results used for final data reporting must be between the low standard and the high standard. Results, which fall below the low standard or above the high

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standard, are to be reported as estimated values. Corrective actions are described in Section 11.11.

10.14 MDLs are determined in reagent water or organic-free sand/soil and verified annually. (Project-specific requirements may require that the MDL study be performed in the site-specific matrix.). See SOP 110_0013 for detailed MDL instructions.

11.0 DATA VALIDATION

The items shall be verified and documented using the VOA Analysis Map in Attachment B.

11.1 Ensure that the BFB tune was run at the beginning of each 12-hour sequence for each batch of samples analyzed. The acceptance criteria are listed below:

BFB Key Ions and Ion Abundance Criteria

Mass	Ion Abundance Criteria
50	15.0-40.0% of mass 95
75	30.0-60.0% of mass 95
95	base peak, 100% relative abundance
96	5.0-9.0% of mass 95
173	< 2.0% if mass 174
174	> 50.0% of mass 95
175	5.0-9.0% of mass 174
176	> 95.0%, but less than 101% of mass 174
177	5.0-9.0% of mass 176

11.1.1 If the BFB acceptance criteria are not met, perform any or all of the following corrective actions:

- Re-purge BFB.
- Retune with PFTBA, and then re-inject BFB.
- Clean MS source, retune with PFTBA, and re-inject BFB.

11.1.2 If the tuning criteria still cannot be met after performing the above, have the mass spectrometer serviced by manufacturer representative.

11.2 After a seven-point initial calibration curve is analyzed, ensure that the following criteria were met.

11.2.1 For the RFs, the %RSD must be less than 30% for all method target analytes, except for the poor purgers where the %RSD must be less than 50%.

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11.2.1.1 If the %RSD of any method target analyte is 15% or less, then the relative response factor is assumed to be constant over the calibration range, and the average relative response factor may be used for quantitation.

11.2.1.2 If the %RSD of any method target analyte is greater than 15%, calibration curves must be constructed using first or higher order regression fits of the six calibration points. ***NOTE: A minimum of 5 points are used for average and linear regression calibration, six point for second order, and seven points for third order calibrations.*** The corresponding Coefficients of Determination (R^2) must be 0.99 or greater.

11.2.1.3 If these acceptance criteria are not met, then the following corrective actions should be performed: (1) adjust the instrument and/or perform instrument maintenance; or (2) narrow the calibration range using standards at different concentrations. The low end of the calibration curves must be carefully watched.

11.2.2 System Performance Check Compounds (SPCCs) and continuing calibration compounds (CCCs) must be evaluated and passed for initial calibration. Refer to Section 8.1.9 and 8.1.10.

11.3 If a 7-point initial calibration was performed, verify that initial calibration verification (ICV) was performed. The percent difference for the CCC analytes must be within 20% (30% for all other target analytes). ***Samples that are to be analyzed for DoD must have ≤20% drift for all compounds, no allowances.***

11.4 After the continuing calibration verification (CCV) standard is analyzed, ensure it was run at the required frequency (every 12 hours, 20 samples, or initially before daily analysis). In addition, the following acceptance criteria must be met.

11.4.1 System Performance Check Compounds (SPCCs): A system performance check must be made during every 12-hour shift. This is the same check that is applied during the initial calibration. If the minimum response factors are not met, the system must be evaluated, and corrective action must be taken before sample analysis begins. The minimum RF for volatile SPCCs is listed in Section 8.1.9.

11.4.2 Calibration Check Compounds (CCCs): After the system performance check is met, CCCs listed in Section 8.1.10 are used to check the validity of the initial calibration with a limit of <20% drift or difference. The acceptance criterion is < 30% drift or difference for all other method target analytes. ***When samples are to be to be run for DoD all compounds must be within <20%D.***

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If these criteria are exceeded, corrective action is necessary. If corrective action fails to produce consecutive (immediate) calibration verification within acceptance criteria, then the analyst may perform maintenance to try to troubleshoot the instrument. After maintenance, the analyst must analyze two consecutive CCV's at two concentrations (one at or below mid-range) to demonstrate that corrective action was successful. If not, then a new six-point calibration must be generated. These criteria must be met before sample analysis begins and re-analysis of samples up to the last acceptable CCV standard must occur with the following exceptions:

- When the acceptance criteria is exceeded high, high bias, then all samples that are non-detects may be reported.

- When the acceptance criteria for the CCV are exceeded low, low bias, sample results that exceed a maximum regulatory limit may be reported.

11.4.3 When performing analyses for Mass DEP MCP, CT-RCP or DoD, any analyte outside of criteria in the CCV must be noted in the project narrative.

11.5 Assess the method blanks. The analyst shall confirm that the method blank was analyzed at the required frequency and the criteria in Section 8.2.4 are met. Analytical batches with Method blanks outside acceptance criteria will be re-prepped and re-analyzed with the following exceptions:

11.5.1 Samples that are that are at least twenty times higher than the method blank may be reported.

11.5.2 When the method blank is less than 5% of the regulatory limit associated with the analyte the method blank would be acceptable.

11.5.3 If the analyte is found in the method blank above the ½ MRL but is not in any of the associated samples, no corrective action is needed.

11.5.4 Any results that are reported with method blank contamination must be B-flagged.

11.6 Assess that matrix spike/matrix spike duplicates were analyzed at required frequency (dependent of sample volume provided). Acceptance criteria are that all % Recovery and/or RPD results meet project-established goals. If no project goals are specified, then results must be within the indicated control limits. If these conditions are not met, perform the following corrective actions as appropriate.

11.6.1 If both BS and MS/MSD recoveries are unacceptable, then the entire batch of field and QC samples must be reanalyzed.

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11.6.2 If the MS/MSD is unacceptable, but the BS is acceptable, then a potential matrix effect has been identified. Reasonable attempts must be made to address matrix interference. The client must be notified in the case narrative of the matrix problem.

11.7 Check the surrogate calculations for correctness for all samples, blanks, BS, MS, and MSD. The following acceptance criteria apply to surrogate recoveries.

11.7.1 The surrogate recoveries for all QC samples must be within control limits of 70-130%. If the BS and/or method blank recoveries are outside limits, re-analysis must be performed for verification. If still outside limits, then corrective action is necessary. ***When samples are to be to be run for the Army Corp of Engineers, surrogate limits must be within 25% for QC samples and 30% for project samples. See Table 3 for surrogate acceptance limits for DoD/Navy/AFCEE projects.***

11.7.2 If sample surrogate recoveries exceed these limits, the sample must be re-analyzed. If the results are still outside the limits, a matrix affect is verified and results need to be discussed in case narrative. ***NOTE: If an obvious matrix effect is displayed on the chromatogram (Unresolved complex mixture, UCM), then re-analysis is not necessary. MA-MCP and CT-RCP projects must have a copy of the chromatogram included with the report.***

11.8 The relative retention times must be checked for all identified compounds in both standards and samples. The internal standard absolute retention times must also be checked for all analyses. Acceptance criteria are as follows:

11.8.1 The relative retention times of each compound in each calibration run should agree within 0.06 relative retention time (RRT) units. Late-eluting compounds usually have much better agreement.

11.8.2 Internal standard retention time:

11.8.2.1 If the retention time for any CCV internal standard changes by more than 30 seconds from the mid-point standard of the initial calibration, the chromatographic system must be inspected for malfunctions and corrections must be made, as required.

11.8.2.2 If the retention time for any sample internal standard changes by more than 30 seconds from the daily calibration, the chromatographic system must be inspected for malfunctions and corrections must be made, as required

11.9 The analyst must verify that ion abundance meets specific criterion for the various analyses. The following acceptance criteria shall be checked for all appropriate samples.

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11.9.1 All ions present in the standard mass spectrum at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) must be present in the sample spectrum.

11.9.2 The relative intensities of ions specified in Section 11.10.1 must agree within plus or minus 30% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample abundance must be between 20 and 80 percent.)

11.9.2.1 Molecular ions present in the reference spectrum should be present in the sample spectrum.

11.9.2.2 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.

11.9.2.3 Ions present in the reference spectrum, but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

11.9.3 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. When serving the role as QA (or referee) laboratory, tentatively identified compounds (TICs) are always reported. Computer-generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. For example, the RCRA permit or waste de-listing requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches will the mass spectral interpretation specialist assign a tentative identification. Guidelines for making tentative identification are as follows:

11.9.3.1 Relative intensities of major ions in the reference spectrum (ions > 10% of the most abundant ion) should be present in the sample spectrum.

11.9.3.2 The relative intensities of the major ions should agree within ±30%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 20 and 80%.)

11.9.3.3 Molecular ions present in the reference spectrum should be present in the sample spectrum. Ions present in the sample spectrum but not

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in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.

11.9.3.4 Ions present in the reference spectrum, but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

11.10 The analyst will check the internal standard area counts for all calibration standards, QC samples, and samples for quantitation. If the area for any of the CCV internal standards changes by a factor of two (-50% to +100%) from the mid-point standard of the initial calibration, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. All sample internal standards must be -50 to 200% if their associated CCV. If the internal standard area counts fail this criterion, the following corrective actions should be considered:

11.10.1 Check to ensure there was no error in internal standards preparation or addition. Also, check instrument performance.

11.10.2 If the CCV failed criterion, re-analyze once. If this second consecutive CCV still does not meet criterion, then a new initial calibration must be performed.

11.10.3 If the internal standards for samples are outside criterion, then re-analyze once. If re-analysis produces results within criteria, then report these results. If the re-analysis is still outside criterion, discuss matrix issue in project narrative. ***NOTE: If an obvious matrix effect is displayed on the chromatogram (Unresolved complex mixture, UCM), then re-analysis is not necessary. MA-MCP and CT-RCP projects must have a copy of the chromatogram included with the report.***

11.11 The analyst must verify all reported results are derived from analytical results that are below the highest standard of the initial calibration curve and above the low standard. Values reported below the low standard are to be reported as estimated values (J values). For samples that exceed the calibration curve, dilute and analyze an appropriate sample aliquot.

11.12 All manual integration must be printed, when made, for verification. See manual integration policy in SOP 110_0016.

11.13 Besides the items listed in Sections 11.1 through 11.12, the analyst should also verify the additional items as noted in Attachment B. A second level of review must be performed by a second analyst; results are recorded on Attachment B.

12.0 REFERENCES

12.1 SW-846 Methods 5035, 8260B and 8000C, Test Methods for the Analysis of Solid Waste, Third Edition, Update III.

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12.2 HP GC EnviroQuant User's Guide, HPG1045A.

12.3 HP Environmental Data Analysis User's Guide HPG0032C.

12.4 HP 6890 GC Operations Manual.

12.5 HP 5890 Series II Operations Manual.

12.6 HP 5971A MS Operations Manual

12.7 HP 5972 MS Operations Manual

12.8 NELAC Standard, Chapter 5, June 2003

12.9 DoD QSM, Version 4.2, October 2010

13.0 ATTACHMENTS

13.1 Attachment A- Run Logbook.

13.2 Attachment B- Analysis Map

13.3 Attachment C- Sample Bottle Prep Procedure for Low Level VOCs in Soil.

13.4 Attachment D- ESS Laboratory VOC 5035 Low Level Sample Collection Procedure.

13.5 Attachment E- Sample Bottle Prep Procedure for High Level VOCs in Soil.

13.6 Attachment F- ESS Laboratory VOC 5035 High Level Sample Collection Procedure.

13.7 Attachment G- Method Summary DQO table.

13.8 Attachment H- Performing Matrix Spike (MS) and Matrix Spike Duplicate Analyses (MSD) for Methanol-Preserved Soil/Sediment Samples under the MCP.

13.9 Attachment J - Encore® sample collection, storage and preparation procedure.

13.10 Attachment K- Mass DEP MCP QC Requirements; Tables II A-1.

14.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

14.1 ESS Laboratory's policies and pollution prevention and waste management are covered in SOP 90_0002, Hazardous Waste Contingency and Emergency Response Plan. All employees are trained in the requirements of the SOP.

15.0 METHOD PERFORMANCE

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15.1 Precision and Accuracy data must be generated by all employees before performing this analysis on client samples. The data is generated by analyzing a method blank and four blank spike samples. Acceptance criteria are 70-130% Recovery and %RSD of ≤ 30%.

15.2 The precision and accuracy data in Table 1 were developed using the high level method. Values are in µg/Kg.

16.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

Table 1. Typical Precision and Accuracy data generated 3/15/2005

Analyte	Spk	Avg	%Rec	%RSD	Analyte	Spk	Avg	%Rec	%RSD
Dichlorodifluoromethane	1250	1320	106	4.0	Toluene	1250	1277	102	2.5
Chloromethane	1250	1208	97	2.6	2-Hexanone	6250	6314	101	2.8
Vinyl Chloride	1250	1303	104	3.4	trans-1,3-Dichloropropene	1250	1294	104	2.1
Bromomethane	1250	1232	99	2.1	1,1,2-Trichloroethane	1250	1302	104	2.3
Chloroethane	1250	1260	101	1.5	1,3-Dichloropropane	1250	1286	103	2.1
Trichlorofluoromethane	1250	1321	106	3.6	Tetrachloroethene	1250	1308	105	2.6
Diethyl ether	1250	1325	106	2.3	Dibromochloromethane	1250	1178	94	2.1
Acetone	6250	6191	99	1.8	1,2-Dibromoethane	1250	1288	103	1.6
Carbon Disulfide	1250	1305	104	2.9	1-Chlorohexane	1250	1253	100	3.5
1,1-Dichloroethene	1250	1304	104	2.7	Chlorobenzene	1250	1267	101	2.2
Methylene Chloride	1250	1279	102	2.3	1,1,1,2-Tetrachloroethane	1250	1292	103	1.9
Methyl tert-Butyl Ether	2500	2618	105	4.3	Ethylbenzene	1250	1255	100	2.2
trans-1,2-Dichloroethene	1250	1297	104	2.6	Xylene P,M	2500	2514	101	1.9
1,1-Dichloroethane	1250	1276	102	2.2	Xylene O	1250	1276	102	2.0
Vinyl Acetate	1250	1250	100	1.6	Styrene	1250	1287	103	2.3
Di-isopropyl ether	1250	1263	101	2.0	Bromoform	1250	1158	93	2.1
Ethyl tertiary-butyl ether	1250	1269	102	1.4	Isopropylbenzene	1250	1275	102	2.3
2-Butanone	6250	6318	101	2.0	Bromofluorobenzene	1250	1138	91	1.9
cis-1,2 Dichloroethene	1250	1286	103	1.7	1,2,3-Trichloropropane	1250	1257	101	2.4
2,2-Dichloropropane	1250	1237	99	3.7	Bromobenzene	1250	1292	103	2.2
Bromochloromethane	1250	1308	105	1.7	1,1,2,2-Tetrachloroethane	1250	1290	103	1.8
Tetrahydrofuran	1250	1231	98	2.7	n-Propylbenzene	1250	1255	100	4.9
Chloroform	1250	1264	101	2.2	2-Chlorotoluene	1250	1265	101	2.7
1,1,1-Trichloroethane	1250	1293	103	3.0	4-Chlorotoluene	1250	1230	98	2.5
1,1-Dichloropropene	1250	1242	99	2.2	1,3,5-Trimethylbenzene	1250	1269	102	2.7
Carbon Tetrachloride	1250	1299	104	3.3	tert-Butylbenzene	1250	1263	101	2.7
Benzene	1250	1278	102	1.9	1,2,4-Trimethylbenzene	1250	1264	101	2.4
1,2-Dichloroethane-d4	1250	1230	98	1.8	sec-Butylbenzene	1250	1262	101	3.1
1,2-Dichloroethane	1250	1278	102	1.7	1,3 Dichlorobenzene	1250	1233	99	2.8
Tertiary-amyl methyl ether	1250	1264	101	2.1	4-Isopropyltoluene	1250	1253	100	2.3

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Trichloroethene	1250	1217	97	1.9	1,4 Dichlorobenzene	1250	1225	98	2.4
1,2-Dichloropropane	1250	1264	101	1.6	n-Butylbenzene	1250	1263	101	2.8
Dibromomethane	1250	1273	102	1.4	1,2 Dichlorobenzene	1250	1263	101	1.7
1,4-Dioxane	25000	23525	94	1.6	1,2-Dibromo-3-Chloropropane	1250	1156	92	2.4
Bromodichloromethane	1250	1287	103	1.8	1,2,4-Trichlorobenzene	1250	1218	97	1.7
4-Methyl-2-Pentanone	6250	6580	105	1.6	Hexachlorobutadiene	1250	1285	103	2.4
cis-1,3-Dichloropropene	1250	1285	103	1.9	Naphthalene	1250	1174	94	2.1
Toluene-d8 (SURR)	1250	1171	94	2.2	1,2,3-Trichlorobenzene	1250	1205	96	2.9

17.0 DEFINITIONS

17.1 **Accuracy**: The closeness of agreement between an observed value and an accepted reference value. When applied to a set of observed values, accuracy will be a combination of a random component and of a common systematic error (or bias) component.

17.2 **Batch**: A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 20, then each group of 20 samples or less will all be handled as a separate batch.

17.3 **Bias**: The deviation due to matrix effects of the measured value (x_s - x_u) from a known spiked amount, where x_s is the spiked sample and x_u is the un-spiked sample. Bias can be assessed by comparing a measured value to an accepted reference value in a sample of known concentration or by determining the recovery of a known amount of contaminant spiked into a sample (matrix spike).

17.4 **Control Sample**: A QC sample introduced into a process to monitor the performance of the system.

17.5 **Equipment Blank**: A sample of analyte-free media which has been used to rinse the sampling equipment. It is collected after completion of decontamination and prior to sampling. This blank is useful in documenting adequate decontamination of sampling equipment.

17.6 **Method Reporting Limit**: The lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The MRL is generally 5 to 10 times the MDL. ESS Laboratory sets the MRL to the lowest non-zero standard in the calibration curve or higher.

17.7 **Field Duplicates**: Independent samples which are collected as close as possible to the same point in space and time. They are two separate samples taken from the same source, stored in separate containers, and analyzed independently. These duplicates are useful in documenting the precision of the sampling process.

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17.8 **Blank Spike (BS)**: A known matrix spiked with compound(s) representative of the target analytes. This is used to document laboratory performance.

17.9 **Matrix**: The component or substrate (e.g., surface water, drinking water) which contains the analyte of interest.

17.10 **Matrix Duplicate**: An intralaboratory split sample which is used to document the precision of a method in a given sample matrix.

17.11 **Matrix Spike**: An aliquot of sample spiked with a known concentration of target analyte(s). The spiking occurs prior to sample preparation and analysis. A matrix spike is used to document the bias of a method in a given sample matrix.

17.12 **Matrix Spike Duplicates**: Intralaboratory split samples spiked with identical concentrations of target analyte(s). The spiking occurs prior to sample preparation and analysis. They are used to document the precision and bias of a method in a given sample matrix.

17.13 **Method Blank**: An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank should be carried through the complete sample preparation and analytical procedure. The method blank is used to document contamination resulting from the analytical process.

17.14 **Method Detection Limit (MDL)**: The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix type containing the analyte. See SOP 110_0013 for further explanation.

17.15 **Organic-Free Reagent Water**: For volatiles, all references to water in the method refer to water in which an interferant is not observed at the method detection limit of the compounds of interest. A water purification system is used to generate organic-free deionized water.

17.16 **Records**: Include all logbooks, papers, machine readable materials, or other documentary materials, regardless of physical form or characteristics.

17.17 **Surrogate**: An organic compound which is similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which is not normally found in environmental samples.

17.18 **Trip Blank**: A sample of analyte-free media taken from the laboratory to the sampling site and returned to the laboratory unopened. A trip blank is used to document contamination attributable to shipping and field handling procedures. This type of blank is useful in documenting contamination of volatile organics samples.

18.0 PERSONNEL QUALIFICATIONS

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18.1 Analysts who perform this analysis must have a working knowledge or quantitative and qualitative analysis, instrumental methods of analysis, chemical laboratory methods, and equipment.

18.2 All analysts, before performing any analysis, participate in the ESS Laboratory training program (SOP80_0016). The training process consists of reading the Standard Operating Procedure, gaining instruction on the procedure from an experienced analyst, and performing the initial demonstration of capability.

19.0 TROUBLESHOOTING

19.1 If BFB criteria are not met, the analysis must be repeated. Analysis can not begin until BFB meets acceptance criteria. Repeated failures indicate that the MS acquisition parameters must be adjusted. These parameters should be adjusted in manual tune, saved and the tune repeated.

19.2 If manual or auto-tune does not produce BFB spectra within acceptance criteria, the MS source may need cleaning. Cleaning instructions are found in the MS detector manual.

19.3 Method blanks must not contain any target compound greater than ½ the MRL with the exception of methylene chloride (see section 11.5 for expanded criteria). If the method blank does not meet criteria, then system must be cleaned before processing samples. This includes washing the purge vessel and baking the trap.

19.4 See laboratory supervisor or operations manager for all other maintenance problems.

19.5 Record all maintenance in the instrument's maintenance logbook.

20.0 DATA MANAGEMENT AND RECORDS

20.1 **Data Management** - ESS Laboratory's utilizes the Promium Element LIMS system as part of its Data Management system. Client sample information is entered into ELEMENT LIMS and analyses are assigned to each sample. The LIMS allows EPA hold times, minimum batch QC requirements, and QC criteria to be assigned to each analysis. Standards can be entered and assigned to QC samples through the LIMS. Once analysis has been performed, data is imported using DataTool avoiding manual errors. In conjunction with Crystal Reports, the ELEMENT system allows for a wide variety of reporting formats.

20.2 **Records** – The specific retention periods required in the NELAC Standards, EPA-CFR and state and local statutes are followed or exceeded. At a minimum, data records are retained for five years from last use (10 years for drinking water). If there is a question about whether a record should be retained or disposed because no specific requirement could be found, the record is retained until such time as a retention period is specified. Records are stored in specified-labeled locations and are easily retrievable. All raw data associated with testing is also retained including; computer printouts, chromatograms, review forms, and logbooks.

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TABLE 2- Internal Standards with their associated compounds

PENTAFLUOROBENZENE (IS)	CHLOROBENZENE-d5 (IS)	1,4-DICHLOROBENZENE-d4 (IS)
Dichlorodifluoromethane	Toluene - d_8	Isopropylbenzene
Chloromethane	Toluene	trans-1,4-Dichloro-2-Butene
Vinyl Chloride	2-Hexanone (P)	1,2,3-Trichloropropane
Bromomethane	Ethyl methacrylate	Bromobenzene
Chloroethane (P)	1,3-Dichloropropane	1,1,2,2-Tetrachloroethane
Trichlorofluoromethane	Tetrachloroethene	n-Propylbenzene
Diethyl Ether	Dibromochloromethane	2-Chlorotoluene
Acrolein (P)	1,2-Dibromoethane	4-Chlorotoluene
1,1,2-Trichloro-1,2,2-trifluoroethane	1-Chlorohexane	1,3,5-Trimethylbenzene
Acetone (P)	Chlorobenzene	tert-Butylbenzene
Iodomethane (P)	1,1,1,2-Tetrachloroethane	Pentachloroethane
Carbon Disulfide (P)	Ethylbenzene	1,2,4-Trimethylbenzene
1,1-Dichloroethene	Xylene, M,O	sec-Butylbenzene
Allyl Chloride	Xylene P	1,3-Dichlorobenzene
Methyl acetate	Styrene	4-Isopropyltoluene
Methylene Chloride	Bromoform	1,4-Dichlorobenzene
Methyl tert-butyl ether	cis 1,4-Dicchloro-2-butene	n-Butylbenzene
Acrylonitrile (P)	4-Bromofluorobenzene*	1,2-Dichlorobenzene
trans-1,2-Dichloroethene		Hexachloroethane
1,1-Dichloroethane		1,2-Dibromo-3-chloropropane (P)
Vinyl Acetate		1,2,4-Trichlorobenzene
Chloroprene		Hexachlorobutadiene
Di-isopropyl ether		Naphtalene
Ethyl tertiary butyl ether		1,2,3-Trichlorobenzene
2-Butanone (P)		
cis-1,2-Dichloroethene		
2,2-Dichloropropane		
Methyl acrylate		
Bromochloromethane		
Methacrylonitrile		
Tetrahydrofuran		
Chloroform		
Dibromofluoromethane*		
1,1,1-Trichloroethane		
Cyclohexane		
1-Chlorobutane		
1,1-Dichloropropene		
Carbon Tetrachloride		
1,2-Dichloroethane-D4*		
Benzene		
1,2-Dichloroethane		
Tertiary-amyl methyl ether		
Trichlorethene		
Methylcyclohexane		
1,2-Dichloropropane		
Dibromomethane		
1,4-Dioxane (P)		
Bromodichloromethane		
2-Nitropropane		
2-Chloroethyl vinyl ether (P)		
4-Methyl-2-pentanone (P)		
cis-1,3-Dichloropropene		
Toluene		
trans-1,3-Dichloropropene		
1,1,2-Trichloroethane		

IS = Internal Standard; * = Surrogate; (P) = Poor purger

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Table 3 (DoD Quality Systems Manual version 4, Table G-5) Blank Spike QC Limits

	DoD			
Analyte	LCL	UCL	LMEL	UMEL
1,1,1,2-Tetrachloroethane	75	125	65	135
1,1,1-Trichloroethane	70	135	55	145
1,1,2,2-Tetrachloroethane	55	130	40	145
1,1,2-Trichloroethane	60	125	50	140
1,1-Dichloroethane	75	125	65	135
1,1-Dichloroethene	65	135	55	150
1,1-Dichloropropene	70	135	60	145
1,2,3-Trichlorobenzene	60	135	50	145
1,2,3-Trichloropropane	65	130	50	140
1,2,4-Trichlorobenzene	65	130	55	140
1,2,4-Trimethylbenzene	65	135	55	145
1,2-Dibromo-3-Chloropropane	40	135	25	150
1,2-Dibromoethane	70	125	60	135
1,2-Dichlorobenzene	75	120	65	125
1,2-Dichloroethane	70	135	60	145
1,2-Dichloropropane	70	120	65	125
1,3,5-Trimethylbenzene	65	135	55	145
1,3 Dichlorobenzene	70	125	65	135
1,3-Dichloropropane	75	125	70	130
1,4 Dichlorobenzene	70	125	65	135
2,2-Dichloropropane	65	135	55	145
2-Butanone	30	160	10	180
2-Chlorotoluene	70	130	60	140
2-Hexanone	45	145	30	160
4-Chlorotoluene	75	125	65	135
4-Methyl-2-Pentanone	45	145	30	165
Acetone	20	160	10	180
Benzene	75	125	65	135
Bromobenzene	65	120	55	130
Bromochloromethane	70	125	60	1358
Bromodichloromethane	70	130	60	135
Bromoform	55	135	45	150
Bromomethane	30	160	10	180
Carbon Disulfide	45	160	30	180
Carbon Tetrachloride	65	135	55	145
Chlorobenzene	75	125	65	130
Dibromochloromethane	65	130	55	140
Chloroethane	40	155	20	175
Chloroform	70	125	65	135

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Chloromethane	50	130	40	140
cis-1,2 Dichloroethene	65	125	55	135
cis-1,3-Dichloropropene	70	125	65	135
Dibromomethane	75	130	65	135
Dichlorodifluoromethane	35	135	15	155
Ethylbenzene	75	125	65	135
Hexachlorobutadiene	55	140	40	155
Isopropylbenzene	75	130	70	140
m,p-Xylene	80	125	70	135
Methylene Chloride	55	140	40	155
Methyl tert-Butyl ether	-	-	-	-
Naphthalene	40	125	25	140
n-Butylbenzene	65	140	50	150
n-Propylbenzene	65	135	50	145
o-Xylene	75	125	70	135
p-Isopropyltoluene	75	135	65	140
sec-Butylbenzene	65	130	50	145
Styrene	75	125	65	135
tert-Butylbenzene	65	130	55	145
Tetrachloroethene	65	140	55	150
Toluene	70	125	60	135
trans-1,2-Dichloroethene	65	135	55	145
trans-1,3-Dichloropropene	65	125	55	140
Trichloroethene	75	125	70	130
Trichlorofluoromethane	25	185	10	215
Vinyl Chloride	60	125	45	140
Surrogates:				
Dibromofluoromethane	85	115	-	-
Toluene-d8	85	115	-	-
4-Bromofluorobenzene	85	120	-	-
1,2-Dichloroehane-d4	75	125	-	-

LCL = Lower Control Limit
UCL = Upper Control Limit
LMEL = Lower Marginal Exceedance Limit
UMEL = Upper Marginal Exceedance Limit

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Attachment A

ESS LABORATORY MS-1 RUN LOG

BATCH DATE	VIAL #	FILE #	LAB ID	METHOD	COMMENTS / DILUTION /STANDARD ID	ANALYST
		M1				
		M1				
		M1				
		M1				
		M1				
		M1				
		M1				
		M1				
		M1				
		M1				
		M1				
		M1				
		M1				
		M1				

Surrogate:_____

On-column IS:_____

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Attachment B

VOA Analysis Map

Analysis _____ **ESS Work Order/Samples** _____

ESS Sample ID	Instr	Directory[1]	File #	Dilt	Batch	Sequence	Cal #	Man Integ'n-Init/Date	RPT	Comments

Comments[2]: _____

VOC Spike ID:_____ **Spike Amount**_____ **Final Volume:**_____

Analyst:_____ **Date:**_____ **Reviewed by:**_____ **(second review includes a check and approval of manual integrations)**

1. Q:/ VOA/"instr"/ 2. All QC compliant unless otherwise noted.

Control #20.0027-0901

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Attachment C

Sample Bottle Prep Procedure for Low Level VOC's in Soil (Method 5035)

Number Of Vials Per Sample: 2 preserved + 1 non-preserved
(Each batch of samples must also include a trip blank)

Preserved Vials:

1) Obtain two VOA vials for each sample.

2) Add the following to each VOA vial:

   a. A small stir bar
   b. 5 ml of organic free water.

3) Cap the vials

4) Place a label on each vial.

5) Label each preserved vial "LL 5035"

6) Weigh each capped and labeled vial.

7) Record the weight of each vial in the appropriate logbook.

8) Assign a tracking number to each vial.

Non-Preserved Vial:

1) Obtain a clean VOA vial.

2) Attach a label and cap.

3) Label vial "% Solids"

Trip Blank: A trip blank consists of a preserved vial labeled "Trip Blank".

Other Equipment:

1) A cut 10 ml syringe should accompany each sample. (Not each vial)

2) The following is supplied if requested:

   a. An EnCore sampler to accompany each preserved vial.
   b. A cooler.
   c. C.O.C's
   d. Ice Packs

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Attachment D

ESS Laboratory VOC 5035 Low Level Sample Collection Procedure

- Number of Vials: **2 per sample**
- **Vial Description:** Pre-weighed VOA vial with 5ml water and a stir bar.
- **Sampling Device:** One Cut Syringe per sample location
- **Sampling Procedure:**
  - Push the cut syringe into the soil until the syringe is filled between the 4 and 5 ml marks. (This is approximately 5 grams of soil)
  - Remove the syringe containing the soil.
  - Wipe the exterior of the syringe clean.
  - Use the plunger to deposit the contents of the syringe into the preserved VOA vial.
  - Immediately cap the vial.
  - Use a new cut syringe for each new sample location.
- **Sample Shipping:**
  - Samples must be kept cool with ice or ice packs ($4 \pm 2^{\circ}$ C)
  - A Low Level **Trip Blank** must accompany each batch of low-level samples. A Trip Blank is a VOA vial with 5mls water and a stir bar. No soil is added.
  - Samples must be frozen upon receipt at laboratory

For additional information call ESS Laboratory: 401–461-7181

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Attachment E

Sample Bottle Prep Procedure for High Level VOC's in Soil (Methanol) (Method 5035)

Number Of Vials Per Sample: 1 preserved + 1 non-preserved (Each batch of samples must also include a trip blank)

Preserved Vials:

- Obtain a VOA vial for each sample.
- Add 15 ml of VOA grade methanol to each VOA vial.
- Cap the vial
- Place a label on each vial.
- Label each preserved vial "5035 MeOH"
- Weigh each capped and labeled vial.
- Record the weight of each vial in the appropriate logbook.
- Assign a tracking number to each vial.
- Make a mark on each preserved vial to indicate where the methanol level should be when approximately 15 grams of soil is added.

Non-Preserved Vial:

- Obtain a clean VOA vial.
- Attach a label and cap.
- Label vial "% Solids"

Trip Blank: A trip blank consists of a preserved vial labeled "Trip Blank".

Other Equipment:

The following is supplied if requested:

- Cooler.
- Chain of custody forms (COC)
- Ice Packs

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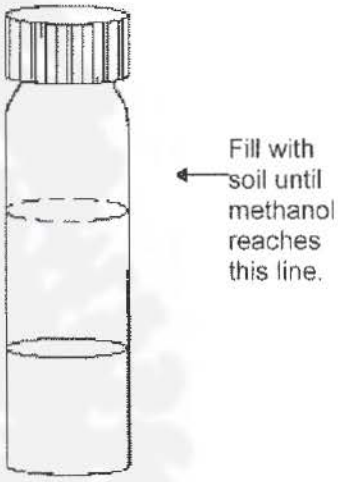
Attachment F

ESS Laboratory VOC 5035
High Level Sample Collection Procedure

- Number of Vials: **1 per sample**
- **Vial Description:** Pre-weighed VOA vial with 15ml methanol and a meniscus mark.
- **Sampling device:** Scoopula or similar spatula
- **Sampling Procedure:**
  - Use the scoopula to add approximately 15 grams of soil to the vial. When approximately 15 grams of soil is in the vial the level of methanol will be even with the drawn meniscus mark.
  - Wipe off any soil that may be on the threads of the vial then immediately cap the vial.



- **Sample Shipping:**
  - Samples must be kept cool with ice or ice packs ($4 \pm 2^{\circ}C$)
  - A High Level **Trip Blank** must accompany each batch of high level samples. A Trip Blank is a VOA vial with 15mls methanol without soil.

Percent Solids Determination

- The percent solids must to be determined for each sample location.
- **Vial Description: VOA** vial with no preservative.
- **Sampling Device:** Scoopula or similar spatula

Sampling Procedure: Use the scoopula to fill the VOA vial ¾ full. Immediately cap the vial.

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Attachment G

Summary of Method Quality Objectives for Method 5035/8260B5
Volatile Organic Compounds

QC Element	Frequency	Target Analyte/Surrogate	Corrective action
Tune	Every twelve hours	Criteria in Section 11.1.	Suspend analysis until criteria met.
Initial Calibration	Instrument set up. Each time the ICV or CCV can not meet criteria.	RSD≤15%, R≥0.995,R^2≥0.99 Do not force through origin	Recalibrate as required.
ICV	Immediately following initial calibration. Second source from Ical	%Rec = 70-130% ***DoD : 75/125%***	Re-analyze once, if still fails criteria re-calibrate.
CCV	At the beginning of each analytical sequence. No more than 20 samples or 12 hours between CCVs.	CCCs must be < 20%, all other analytes < 30% drift or difference. ***DoD : 80-120% all compounds.***	Re-analyze once, if still fails criteria re-calibrate.
Blank spike/ Blank spike duplicate	At the beginning of each analytical sequence. Prepared using source different than that used to prepare initial calibration standards and contain all target analytes.	%Rec = 70-130% ≤ 25% drift or difference. ***See Table 3 for DoD criteria***	Re-analyze once, if still fails criteria re-calibrate.
Method Blank	One per analytical batch of 20 or fewer samples.	Analytes < ½ MRL (DCM≤ MRL)	See Section 11.0. Report nonconformance in case narrative.
MS/MSD	1 per 20 samples. Should be spiked with the same solution as the BS.	% Rec = 70-130%, ≤ 30% RPD ***See Table 3 for DoD criteria***	Check BS, if BS is within criteria then report non-conformance in project narrative. If BS fails, then re-analyze.
Surrogates	Added to all samples and standards.	%Rec = 70-130% ***See Table 3 for DoD criteria***	If one or more is outside criteria, then re-analyze sample unless an obvious interference is present (UCM).
Internal Standards	Added to all samples and standards.	50-200% of area counts in the associated continuing calibration standard.	If one or more is outside criteria, then re-analyze sample unless an obvious interference is present (UCM).

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Attachment H

Performing Matrix Spike (MS) and Matrix Spike Duplicate Analyses (MSD) for Methanol-Preserved Soil/Sediment Samples under the MCP

a. An unpreserved aliquot of soil/sediment sample must be thoroughly homogenized.

b. Place a 5 g aliquot of homogenized soil in each of three (3) pre-tared 40-ml VOA vials. Record the weights of the vials with and without sample in the VOA sample weight logbook.

c. Cap each vial ensuring that soil particles are not entrained on the vial threads.

d. Without exposing the sample aliquot to the atmosphere, prepare an un-spiked sample, designated **C0**, by adding 5 ml of purge and trap grade methanol through the septum of the vial. Tap and agitate the sample vial.

Note: This sample may only be used to determine the residual concentration in the laboratory-homogenized sample and is not representative of the contaminant concentration at the location where the field sample was collected.

e. Without exposing the sample aliquot to the atmosphere, prepare two (2) separate spiked samples, designated **Cx1** and **Cx2**, by adding 0.5 ml of the MS standard to each of the remaining pre-tared VOA vials.

f. Allow vials **Cx1** and **Cx2** to equilibrate for at least 30 minutes. After the equilibration period, bring the final solvent volume in vials **Cx1** and **Cx2** up to 5ml by adding purge and trap grade methanol through the septum of each vial.

g. Proceed with analysis for high-level VOCs using methanol preservation. **Vials may not be opened until soils are completely immersed in methanol.**

h. Record the results for **C0** (unspiked sample), **Cx1** (method spike), and **Cx2** (method spike duplicate). Calculate and report the RPD and %R for the subject sample matrix. It should be noted that this procedure might result in the loss of VOCs from the native sample during the homogenization step. However, the intent of this procedure is solely to determine matrix effects and not to measure the actual concentrations present in the native sample.

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Attachment J

ESS Laboratory VOC 5035

Encore Sample Collection, Storage and Preparation Procedure

Encore™® or Equivalent Sampling Device for low or high level analysis: Method 5035 contains an option with a proprietary device called an Encore™® sampler. This is a special, one use, EPA approved sampling device that allows the sampler to collect and transport samples to the laboratory without preserving in the field. The Encore™® sampler is a device made of an inert composite polymer, designed to collect, store and deliver soil in a sealed, headspace-free state. In order to collect the sample the Encore™® sampler is attached to a reusable T-handle that serves to assist in pushing the sampler in the soil. An airtight sealing cap is then attached creating a self-contained package. Generally, 3 Encore™® samplers are required for each sample providing the laboratory with 2 for low level analysis and 1 for high level analysis. The devices are transported to the laboratory on ice where the cap is opened and the undisturbed sample is immediately placed in the appropriate preservation fluid, methanol or low level vials containing water. This preservation step must currently take place within 48 hours of sample collection.

ESS Laboratory has two options for meeting method 5035A sampling and preservation requirements.

Option 1 (The option preferred by ESS Laboratory)
Sample collection will require the following:

- 2 pre-weighed vials containing a small magnetic stir bar and 5mls DI water for low level analysis.
- 1 pre-weighed vial for medium-high level analysis containing 15 mls of methanol.
- 1 four ounce jar for percent total solids determination.
- Methanol is flammable. If samples will be shipped to the laboratory via couriers such as, UPS or Federal Express, DOT labeling requirements must be met. Contact the lab for additional information concerning labeling requirements.

Option 2
Sample collection using *EnCore™* samplers will require:

- 2 EnCore™ 5g samplers for low level analysis
- 1 EnCore™ 5g sampler for medium-high level analysis
- 1 four ounce jar for percent total solids determination
- T-handle for sampling

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Encore Sampling Procedure:

Disposable En Core® Sampler Sampling Procedures

NOTE:

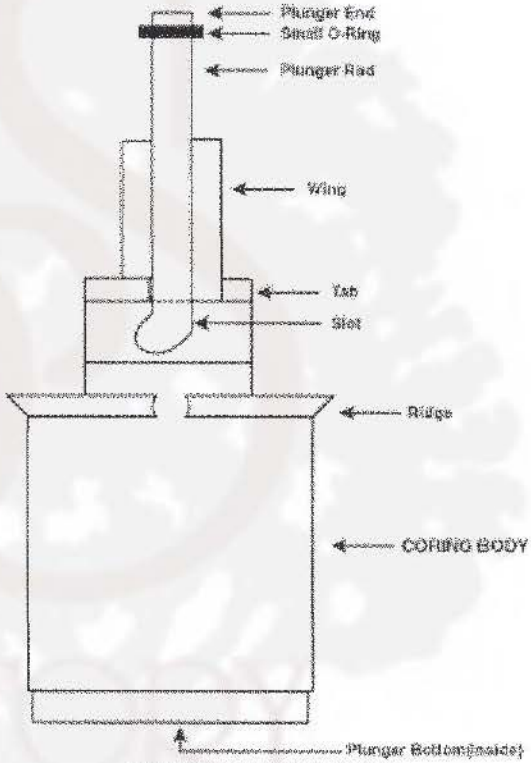
1. En Core Sampler is a Single Use device. It cannot be cleaned and/or reused.

2. En Core Sampler is designed to store soil. Do not use En Core Sampler to store solvent or free product!

3. En Core Sampler must be used with En Core® T-Handle and/or En Core® Extrusion Tool exclusively. (These items are sold separately.)

Using The En Core® T-Handle

Before Taking Sample:
1. Hold coring body and push plunger rod down until small o-ring rests against tabs. This will assure that plunger moves freely.



Click to view larger image.

2. Depress locking lever on En Core T-Handle. Place coring body, plunger end first, into open end of T-Handle, *aligning the (2) slots on the coring body with the (2) locking pins in the T-Handle.* Twist coring body clockwise to lock pins in slots. Check to ensure Sampler is locked in place. Sampler is ready for use.

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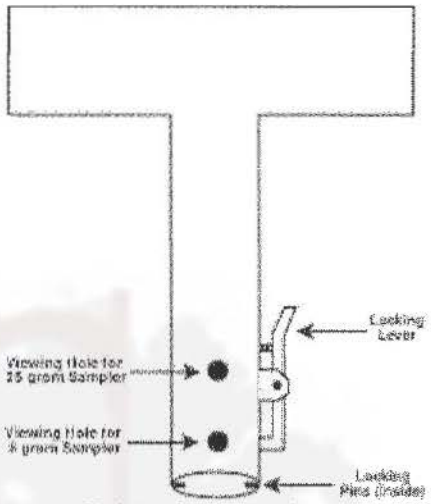
Taking Sample:

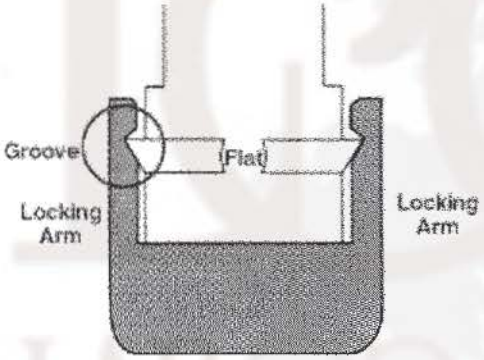
3. Turn T-Handle with T-up and coring body down. This positions plunger bottom flush with bottom of coring body (ensure that plunger bottom is in position). Using T-Handle, push Sampler into soil until coring body is completely full. When full, small o-ring will be centered in T-Handle viewing hole. Remove Sampler from soil. Wipe excess soil from coring body exterior.

4. Cap coring body while it is still on T-handle. Push cap over flat area of ridge. ***Push and twist cap to lock arm in place. Cap must be seated to seal sampler (see diagram below).***

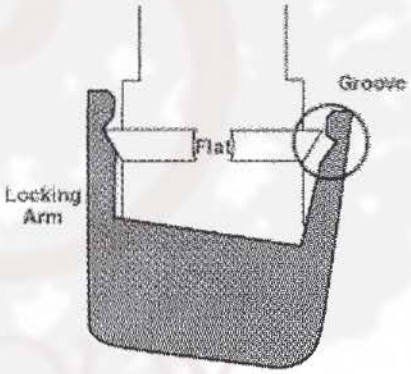


Click to view larger image

Sampler Correctly Capped
Locking arm grooves seated over coring body ridge.



Sampler Incorrectly Capped
Cap appears crooked; locking arm grooves not fully seated over coring body ridge



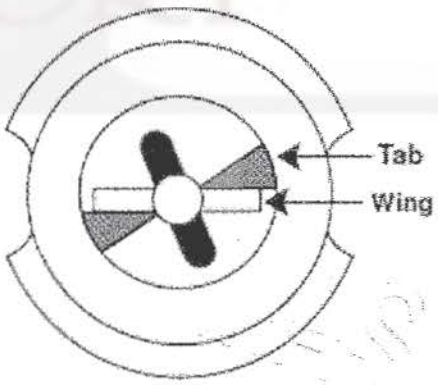
Preparing Sampler For Shipment:

5. Remove the capped Sampler by depressing locking lever on T-Handle while twisting and pulling Sampler from T-Handle.

6. Lock plunger by rotating extended plunger rod fully counter-clockwise until wings rest firmly against tabs (see plunger diagram at right).

7. Attach completed label (from En Core Sampler bag) to cap on coring body.

8. Return full En Core Sampler to zipper bag. Seal bag and put on ice.



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Encore Sample Prep:

Since samples must be preserved within 48 hours of sampling, samples are transferred into preserved vials upon receipt. Laboratory preservation is documented on the cooler receipt checklist.

Sample Extrusion Procedure with Extrusion Tool:

Disposable En Core® Sampler Extrusion Procedure
Using The En Core® Extrusion Tool

- 1. Use a pliers to break locking arms on cap of En Core Sampler. ***Do not remove cap at this time. (CAUTION: Broken edges will be sharp.)***
- 2. To attach En Core Sampler to En Core Extrusion Tool: Depress locking lever on Extrusion Tool and place Sampler, plunger end first, into open end of Extrusion Tool, ***aligning slots on coring body with pins in Extrusion Tool.*** Turn coring body clockwise until it locks into place. Release locking lever.
- 3. Rotate and gently push Extrusion Tool plunger knob clockwise until plunger slides over wings of coring body. (When properly positioned plunger will not rotate further.)
- 4. Hold Extrusion Tool with capped Sampler pointed upward so soil does not fall out when cap is removed. To release soil core, remove cap from Sampler and push down on plunger knob of En Core Extrusion Tool. Remove and properly dispose of En Core Sampler.

The 5 gram soil sample is released into one of the following vials:

1. **Low Level VOC analysis:** 5 grams released into a 40ml VOA vial containing a small stir bar and 5ml DI water. If sample is not being analyzed that day, the vials are stored frozen in a freezer at <5C. Two vials required. One vial is a backup for reanalysis or confirmation.

2. **Med-High Level VOC analysis:** 5 grams released into a 40ml VOA vial containing 5ml of MEOH. If sample is not being analyzed that day, the vials are stored in a refrigerator at 4 +/- 2C. One vial required.

3. **GRO analysis: :** 5 grams released into a 40ml VOA vial containing 5ml of MEOH. If sample is not being analyzed that day, the vials are stored in a refrigerator at 4 +/- 2C. One vial required.

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Attachment K

Quality Control Requirements and Performance Standards for the ***Analysis of Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)*** in Support of Response Actions under the Massachusetts Contingency Plan (MCP)

Table II A-1: Specific QC Requirements and Performance Standards for VOCs (SW-846 8260B) Using WSC-CAM-II A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
Initial Demonstration of Proficiency	Laboratory Analytical Accuracy & Precision	(1) Must be performed prior to using method on samples. (2) Must be performed for each matrix. (3) Must contain all target analytes. (4) Must follow procedure in Section 8.4 of SW-846 8000B.	No	NA	Refer to Section 8.4 of SW-846 8000B and Section 1.1.2 of this protocol.	NA
GC/MS Tunes with BFB	Inter-laboratory Consistency & Comparability	(1) Criteria listed in Table 4 of SW-846 8260B (the same criteria must be used for all analyses). (2) Every 12 hours prior to sample analysis.	No	NA	Perform instrument maintenance as necessary; retune instrument.	Suspend all analyses until tuning non-compliance is rectified.
Initial Calibration	Laboratory Analytical Accuracy	(1) Must be analyzed at least once prior to analyzing samples, when initial calibration verification or continuing calibration does not meet the performance standards, and when major instrument maintenance is performed. (2) Minimum of 5 standards (or 6 if non-linear regression used). (3) Low standard must be ≤RL. (4) %RSD ≤20, r ≥0.99 (linear regression), or r^2 ≥0.99 (non-linear regression) for each target analyte. (5) If %RSD >20, linear or non-linear regression must be used. (6) Minimum RFs as per Table 4 of SW-846 8260C for lowest concentration standard and for average RF. (7) Must contain all target analytes. (8) Calibration must be performed under the same conditions as the samples (e.g., heated purge). (9) If autosampler used to spike surrogates in calibration standards, one-point	No	RF <0.05; affects nondetect results for affected analyte in all samples analyzed under this initial calibration.	(1) Recalibrate if >10% of target analytes exceed %RSD, "r", or "r^2" criteria. (2) If ≤10% of compounds exceed criteria, recalibration is not required as long as %RSD <40, r >0.98, or r^2 >0.98. (3) If recalculated concentrations from the lowest calibration standard are outside of 70-130% recovery range, either: * The RL must be reported as an estimated value[2], or * The RL must be raised to the concentration of the next highest calibration standard that exhibits acceptable recoveries when recalculated using the final calibration curve.	Sample analysis cannot proceed without a valid initial calibration. Report non-conforming compounds (%RSD >20, r <0.99, r^2 <0.99 or minimum RF not met) in laboratory narrative. If non-linear regression (i.e., quadratic equation) is used for calibration, this must be noted in the laboratory narrative along with the compounds affected.

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Table II A-1: Specific QC Requirements and Performance Standards for VOCs (SW-846 8260B) Using WSC-CAM-II A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
		calibration with 5 standards acceptable for surrogates. (10) If linear or non-linear regression used, verify the RL by recalculating concentrations in lowest calibration standard using the final calibration curve; recoveries must be 70-130%. (11) SIM: Laboratory must monitor a minimum of two ions per analyte (the primary ion or quantitation ion and a minimum of one confirmation ion); this is required for all target analytes, surrogates and internal standards.				
Initial Calibration Verification	Laboratory Analytical Accuracy	(1) Immediately after each initial calibration. (2) Concentration level near midpoint of curve. (3) Prepared using standard source different than used for initial calibration. (4) Must contain all target analytes. (5) Percent recoveries must be between 70-130% for target analytes except for "difficult" analytes[**] which must exhibit percent recoveries between 40-160%.	No	NA	Locate source of problem; recalibrate if >10% of all analytes are outside of criteria.	If recovery is outside of 70-130% for any analyte, including "difficult" analytes[**], report non-conforming compounds in laboratory narrative.
Continuing Calibration	Laboratory Analytical Accuracy	(1) Every 12 hours prior to the analysis of samples. (2) Concentration level near midpoint of curve. (3) Must contain all target analytes. (4) %D must be ≤20 for each target analyte. (5) Minimum RFs as per Table 4 of SW-846 8260C. (6) Area counts of internal standards in continuing calibration must be between 50 – 200% of the area counts in the associated mid-level initial calibration	No	RF <0.05; affects nondetect results for affected analyte in all samples analyzed under this continuing calibration.	(1) Recalibrate if >20% of target analytes exceed %D criteria. (2) If internal standard is outside of criteria, locate source of problem and reanalyze the continuing calibration. (3) If ≤20% of compounds exceed criteria, recalibration is not required as long as %D	Report non-conforming compounds (%D >20 or minimum RF not met) and associated samples in laboratory narrative.

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Table II A-1: Specific QC Requirements and Performance Standards for VOCs (SW-846 8260B) Using WSC-CAM-II A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
		standard.			<40.	
Method Blank	Laboratory Method Sensitivity (contamination evaluation)	(1) Every 20 samples prior to sample analysis and after calibration standards. (2) Matrix and preservative-specific (e.g., water, methanol). (3) Target analytes must be <RL except for common laboratory contaminants (acetone, methylene chloride, and MEK) which must be <5x the RL.	Yes	NA	(1) If concentration of contaminant in sample is <10x concentration in blank, locate source of contamination; correct problem; reanalyze method blank and associated samples. (2) No corrective action required if concentration of contaminant in sample is >10x concentration in blank or if contaminant not detected in sample.	(1) If sample reanalysis is not possible, report non-conformance in laboratory narrative. (2) If contamination of method blanks is suspected or present, the laboratory, using a "B" or some other convention, should qualify the sample results. Blank contamination should also be documented in the laboratory narrative. (3) If re-analysis is performed within holding time and yields acceptable method blank results, the laboratory may report results of the re-analysis only. (4) If re-analysis is performed outside of holding time, the laboratory must report results of both the initial analysis and re-analysis.
Laboratory Control Sample (LCS)	Laboratory Analytical Accuracy	(1) Every 20 samples or for each new tune clock, whichever is more frequent.	Yes	Recovery <10%; affects nondetect	(1) Locate source of problem; reanalyze LCS	(1) If sample reanalysis is not possible, report non-

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Table II A-1: Specific QC Requirements and Performance Standards for VOCs (SW-846 8260B) Using WSC-CAM-II A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
		(2) Concentration level near midpoint of curve. (3) Must contain all target analytes. (4) Matrix and preservative-specific (e.g., water, methanol). (5) Percent recoveries must be between 70-130% for target analytes except for "difficult" analytes[**] which must exhibit percent recoveries between 40-160%. (6) Can also be used as continuing calibration. NOTE: If used as continuing calibration standard, must be evaluated using Performance Standards, Corrective Actions, and Analytical Response Actions listed above for Continuing Calibration.		results for affected analyte in all samples analyzed under the LCS.	and associated samples if >10% of all analytes are outside of criteria. (2) If ≤10% of compounds are outside of the acceptance criteria, reanalysis is not required as long as recoveries are >10%. (3) If >10% of compounds are above the acceptance criteria (>130%), reanalysis is not required if affected compounds were not detected in associated samples.	conformance in laboratory narrative. (2) If recovery is outside of 70-130% for any analyte, including "difficult" analytes[**], report non-conforming compounds in laboratory narrative. (3) If re-analysis is performed within holding time and yields acceptable LCS results, the laboratory may report results of the re-analysis only. (4) If re-analysis is performed outside of holding time, the laboratory must report results of both the initial analysis and re-analysis.
LCS Duplicate	Laboratory Analytical Accuracy & Precision	(1) Every 20 samples or for each new tune clock, whichever is more frequent. (2) Concentration level near midpoint of curve. (3) Must contain all target analytes. (4) Matrix and preservative-specific (e.g., water, methanol). (5) Percent recoveries must be between 70-130% for target analytes except for "difficult" analytes[**] which must exhibit percent recoveries between 40-160%. (6) Recommended to be run immediately after LCS in analytical sequence. (7) RPDs must be ≤20 for waters and solid.	Yes	Recovery <10%; affects nondetect results for affected analyte in all samples analyzed under this LCS.	(1) Locate source of problem; reanalyze LCS and associated samples if >10% of all analytes are outside of the recovery acceptance criteria. (2) If ≤10% of compounds are outside of the recovery acceptance criteria, reanalysis is not required as long as recoveries are >10%. (3) If >10% of compounds are above the recovery acceptance criteria (>130%), reanalysis is not	(1) If sample reanalysis is not possible, report non-conformance in laboratory narrative. (2) If recovery is outside of 70-130% for any analyte, including "difficult" analytes[**] or RPD >20 for any analyte, including "difficult" analytes**, report non-conforming compounds in laboratory narrative. (3) If re-analysis is performed within holding

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Table II A-1: Specific QC Requirements and Performance Standards for VOCs (SW-846 8260B) Using WSC-CAM-II A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
					required if affected compounds were not detected in associated samples.	time and yields acceptable LCS results, the laboratory may report results of the re-analysis only. (4) If re-analysis is performed outside of holding time, the laboratory must report results of both the initial analysis and re-analysis.
MS/MSD	Method Accuracy & Precision in Sample Matrix	(1) Every 20 samples (at discretion of laboratory or at request of data user). (2) Matrix-specific. (3) Concentration level near midpoint of curve. (4) Must contain all target analytes. (5) Percent recoveries between 70 – 130%. (6) RPDs ≤20 for waters and ≤30 for solids.	Yes ONLY when requested by the data user	Recovery <10%; affects nondetect result for affected analyte in unspiked sample only.	Check LCS; if recoveries are acceptable in LCS, narrate non-conformance.	Note exceedances in laboratory narrative.
Surrogates	Method Accuracy in Sample Matrix	(1) Minimum of 3 surrogates, at retention times across GC run. (2) Percent recoveries must be between 70-130% for individual surrogate compounds.	Yes	Recovery <10%; affects all nondetect VOC results in affected sample.	If one or more surrogates are outside of limits, reanalyze sample unless one of the following exceptions applies: (1) Obvious interference present (e.g., UCM). NOTE: If obvious interference is present and surrogate recovery would cause rejection of data (i.e., <10%), reanalyze sample on dilution. (2) Methanol-preserved samples: re-analysis is not	(1) Report recoveries outside of 70-130% in laboratory narrative. (2) If reanalysis yields similar surrogate non-conformances, the laboratory must report results of both analyses. (3) If reanalysis is performed within holding time and yields acceptable surrogate recoveries, the laboratory may report results of the reanalysis only.

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Table II A-1: Specific QC Requirements and Performance Standards for VOCs (SW-846 8260B) Using WSC-CAM-II A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
					required if % moisture >25 and surrogate recovery is >10%. (3) If one or more surrogates exhibit high recoveries and target analytes are not detected in sample, reanalysis is not required.	(4) If reanalysis is performed outside of the holding time and yields acceptable surrogate recoveries, the laboratory must report results of both analyses. (5) If sample is not re-analyzed due to obvious interference, the laboratory must provide the chromatogram in the data report.
Internal Standards	Laboratory Analytical Accuracy and Method Accuracy in Sample Matrix	(1) Minimum of 3 at retention times across GC run. (2) Area counts in samples must be between 50 – 200% of the area counts in the associated continuing calibration standard. (3) Retention times of internal standards must be within ±30 seconds of retention times in associated continuing calibration standard.	No	Recovery <20%; affects all nondetect results quantitated using affected internal standard in associated sample.	If one or more internal standards are outside of limits, reanalyze sample unless obvious interference present (e.g., UCM). NOTE: If obvious interference is present and internal standard area would cause rejection of data (i.e., <20%), reanalyze sample on dilution.	(1) Report nonconformances in laboratory narrative. Include actual recovery of internal standard and provide summary of analytes quantitated using the internal standard. (2) If reanalysis yields similar internal standard non-conformances, the laboratory must report results of both analyses. (3) If reanalysis is performed within holding time and yields acceptable internal standard recoveries, the laboratory may report results of the reanalysis only. (4) If reanalysis is performed outside of the holding time and yields

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Table II A-1: Specific QC Requirements and Performance Standards for VOCs (SW-846 8260B) Using WSC-CAM-II A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
						acceptable internal standard recoveries, the laboratory must report results of both analyses. (5) If sample is not reanalyzed due to obvious interference, the laboratory must provide the chromatogram in the data report.
Quantitation	NA	(1) Quantitation must be based on internal standard calibration. (2) The laboratory must use the average response factor, linear or non-linear regression curve generated from the associated initial calibration for quantitation of each analyte. (3) The internal standard used for quantitation must be the one nearest the retention time of the subject analyte. (4) Results must be reported with 2 or more "significant figures" if ≥RL. If reporting values below the RL, report with 1 or more "significant figures".[3]	NA	NA	NA	NA
Identification	NA	Refer to SW-846 8260B, Section 7.6.	NA	NA	NA	NA
General Reporting Issues	NA	(1) The laboratory must only report values ≥ the sample-specific reporting limit; optionally, values below the sample-specific reporting limit can be reported as estimated, if requested. The laboratory must report results for samples and blanks in a consistent manner. (2) Dilutions: If diluted and undiluted analyses are performed, the laboratory	NA	NA	NA	(1) Qualification of the data is required if reporting values below the sample-specific reporting limit. (2) Complete analytical documentation for diluted and undiluted analyses must be made available for review

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Table II A-1: Specific QC Requirements and Performance Standards for VOCs (SW-846 8260B) Using WSC-CAM-II A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
		should report results for the lowest dilution within the valid calibration range for each analyte. The associated QC (e.g., method blanks, surrogates, etc.) for each analysis must be reported. (3) Refer to Section 3.3, TICs by GC/MS, for guidance. (4) All soil/sediment sample results preserved in methanol must be corrected for the methanol dilution as per Section 3.2.1 of this CAM protocol. (5) Results for soils/sediments must be reported on a dry-weight basis for comparison to MCP regulatory standards. (6) Refer to Appendix II A-1 for chain-of-custody requirements regarding preservation, cooler temperature, and holding times.				during an audit. (3) TICs will be evaluated at the discretion of the data user consistent with the guidelines presented in Appendix II A–3. (4) The performance of dilutions must be documented in the laboratory narrative or on the report form. Unless due to elevated concentrations of target compounds, reasons for dilutions must be explained in the laboratory narrative. (5) If samples are not properly preserved (pH >2 for aqueous samples, solid samples not completely covered with appropriate preservative) or are not received with an acceptable cooler temperature, note the non-conformances in the laboratory narrative. (6) If samples are preserved and/or analyzed outside of the holding time, note the non-conformances in the laboratory narrative.

** Potentially "difficult" analytes include: acetone, methyl ethyl ketone, 4-methyl-2-pentanone, 2-hexanone, dichlorodifluoromethane, bromomethane, chloromethane, and 1,4-dioxane.

[1] As per Appendix IV of MassDEP Policy #WSC-07-350, *MCP Representativeness Evaluations and Data Usability Assessments*, September 2007, if these results are observed, data users should consider nondetect results as unusable and positive results as estimated with a significant low bias.

[2] If the RL is estimated due to unacceptable recovery of the lowest standard, the CAM RL has not been achieved; Question G of the "MassDEP MCP Analytical Protocol Certification Form" must be answered

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Table II A-1: Specific QC Requirements and Performance Standards for VOCs (SW-846 8260B) Using WSC-CAM-II A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action

"NO" and this must be addressed in the laboratory narrative.

[3]Reporting protocol for "significant figures" is a policy decision included for standardization and consistency for reporting of results and is not a definition of "significant" in the scientific or mathematical sense.

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Division of Thielsch Engineering
Cranston, RI

SOP NO. 20_8260B
VOLATILE ORGANIC COMPOUNDS
BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS):
CAPILLARY COLUMN TECHNIQUE
(EPA Method 624/SW-846 METHOD 8260B)

APROVED BY:

Operation s Manager — Date 3/20/9

QA Manager — Date 3/20/09

Laboratory Director — Date 3/20/09

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Volatile Organic Compounds - by GC/MS Capillary Column Technique

1.0 SCOPE AND APPLICATION

1.1 This method describes the technique utilized by ESS Laboratory in the qualitative and quantitative analysis of volatile samples based on SW-846 Method 8260B. Following are compounds that may be determined by this method.

Appropriate Technique Analyte	Cas #[b]	Purge-and-Trap	Direct Injection
Acetone	67-64-1	Pp	a
Acrolein (Propenal)	107-02-8	Pp	a
Acrylonitrile	107-13-1	Pp	a
Allyl chloride	107-05-1	a	a
tert- Amyl methyl ether	994-05-8	a	a
Benzene	71-43-2	a	a
Bromobenzene	108-86-1	a	a
Bromochloromethane	74-97-5	a	a
Bromodichloromethane	75-27-4	a	a
4-Bromofluorobenzene	460-00-4	a	a
Bromoform	75-25-2	a	a
Bromomethane	74-83-9	a	a
2-Butanone (MEK)	78-93-3	Pp	a
n-Butylbenzene	104-51-8	a	a
sec-Butylbenzene	135-98-8	a	a
tert-Butylbenzene	98-06-6	a	a
Carbon disulfide	75-15-0	Pp	a
Carbon tetrachloride	56-23-5	a	a
Chlorobenzene	108-90-7	a	a
1-Chlorobutane	109-69-3	a	a
Chlorodibromomethane	124-48-1	a	a
Chloroethane	75-00-3	a	a
2-Chloroethyl vinyl ether	110-75-8	a	a
Chloroform	67-66-3	a	a
Chloromethane	74-87-3	a	a

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Chloroprene	126-99-8	a	a

Appropriate Technique Analyte	Cas #[b]	Purge-and-Trap	Direct Injection
2-Chlorotoluene	95-49-8	a	a
4-Chlorotoluene	106-43-4	a	a
Cyclohexane	110-82-7	a	a
1,2-Dibromo-3-chloropropane	96-12-8	Pp	a
1,2-Dibromoethane	106-93-4	a	a
Dibromomethane	74-95-3	a	a
1,2-Dichlorobenzene	95-50-1	a	a
1,3-Dichlorobenzene	541-73-1	a	a
1,4-Dichlorobenzene	106-46-7	a	a
Cis-1,4-Dichloro-2-butene	1476-11-5	a	a
trans-1,4-Dichloro-2-butene	110-57-6	Pp	a
Dichlorodifluoromethane	75-71-8	a	a
1,1-Dichloroethane	75-34-3	a	a
1,2-Dichloroethane	107-06-2	a	a
1,1-Dichloroethene	75-35-4	a	a
cis-1,2-Dichloroethene	156-59-2	a	a
trans-1,2-Dichloroethene	156-60-5	a	a
1,2-Dichloropropane	78-87-5	a	a
1,3-Dichloropropane	142-28-9	a	a
2,2-Dichloropropane	594-20-7	a	a
1,1-Dichloropropene	563-58-6	a	a
cis-1,3-Dichloropropene	10061-01-5	a	a
trans-1,3-Dichloropropene	10061-02-6	a	a
1,4-Difluorobenzene	540-36-3	a	a
Diethyl Ether	60-29-7	a	a
1,4-Dioxane	123-91-1	Pp	a
Ethylbenzene	100-41-4	a	A
Ethyl methacrylate	97-63-2	a	a
Ethyl tertiary-butyl ether	637-92-3	a	a
Hexachlorobutadiene	87-68-3	a	a
Hexachloroethane	67-72-1	i	a
2-Hexanone	591-78-6	Pp	a

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Appropriate Technique Analyte	Cas #[b]	Purge-and-Trap	Direct Injection
Iodomethane	74-88-4	a	a
Isopropylbenzene	98-82-8	a	a
Di-isopropyl ether	108-20-3	a	a
p-Isopropyltoluene	99-87-6	a	a
Methyl acetate	79-20-9	a	a
Methyl acrylate	96-33-3	a	a
Methylacrylonitrile	126-98-7	pp	a
Methyl Cyclohexane	108-87-2	a	a
Methylene chloride	75-09-2	a	a
Methyl methacrylate	80-62-6	a	a
4-Methyl-2-pentanone (MIBK)	108-10-1	pp	a
Methyl tertiary-butyl ether	1634-04-4	a	a
Naphthalene	91-20-3	a	a
2-Nitropropane	79-46-9	a	a
Pentachloroethane	76-01-7	i	a
n-Propylbenzene	103-65-1	a	a
Styrene	100-42-5	a	a
1,1,1,2-Tetrachloroethane	630-20-6	a	a
1,1,2,2-Tetrachloroethane	79-34-5	a	a
Tetrachloroethene	127-18-4	a	a
Toluene	108-88-3	a	a
1,2,3-Trichlorobenzene	87-61-6	a	a
1,2,4-Trichlorobenzene	120-82-1	a	a
1,3,5-Trichlorobenzene	108-70-3	a	a
1,1,1-Trichloroethane	71-55-6	a	a
1,1,2-Trichloroethane	79-00-5	a	a
Trichloroethene	79-01-6	a	a
Trichlorofluoromethane	75-69-4	a	a
1,2,3-Trichloropropane	96-18-4	a	a
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	a	a
1,2,4-Trimethylbenzene	95-63-6	a	a
1,3,5-Trimethylbenzene	108-67-8	a	a
Vinyl acetate	108-05-4	a	a
Vinyl chloride	75-01-4	a	a
o-Xylene	95-47-6	a	a

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m-Xylene	108-38-3	a	a
p-Xylene	106-42-3	a	a

a Adequate response by this technique.
b Chemical Abstract Services Registry Number.
i Inappropriate technique for this method
pp Poor purging efficiency results in high MDLs.

1.2 This method is used to determine volatile organic compounds in a variety of waste matrices. This method is applicable to nearly all types of samples, including ground water, aqueous sludge, caustic liquors, acid liquors, waste solvents, oily wastes, tars, fibrous wastes, emulsions, filter cakes, soils and sediments.

1.3 This method can be used to quantify most volatile organic compounds that have boiling points below 200°C and that are insoluble or slightly soluble in water. Volatile water-soluble compounds can be included in this analytical technique, however, for the more soluble compounds quantitation limits are approximately ten times higher because of poor purging efficiency.

1.4 The quantitation limit of this method for an individual compound is approximately 1µg/L for all aqueous matrices. The Quantitation limit for soil/sediment samples is listed in SOP 20_5035.

2.0 METHOD SUMMARY

2.1 This SOP provides gas chromatographic/mass spectrometric (GC/MS) conditions for the detection of volatile organic compounds.

2.2 This SOP describes a closed system purge and trap procedure for aqueous samples according to method 5030B.

2.2.1 Purged sample components are trapped in a tube containing suitable sorbent materials.

2.2.2 When purging is complete, the sorbent tube is heated and back-flushed with helium to desorb trapped sample components. The analytes are desorbed directly to a wide bore capillary column for analysis.

2.2.3 Wide-bore capillary columns require a jet separator, whereas narrow bore capillary columns can be directly interfaced to the ion source.

2.3 A temperature program is used in the gas chromatograph to separate the organic compounds. Detection is achieved using a mass spectrometer (MS).

2.4 Tentative identifications are obtained by analyzing standards under the same conditions used for samples and comparing resultant GC retention times. Absolute identifications are obtained by comparing the mass spectra of individual compounds to the reference spectra for that compound.

2.5 Concentrations of the identified components are measured by relating the response produced for that compound to the response produced by a compound that is used

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as an internal standard.

3.0 HEALTH AND SAFETY

3.1 Each employee has been trained and has acknowledged being trained in the safe use and handling of chemicals being used in the laboratory. This training has been performed according to the ESS Training SOP 80_0016 and by the Chemical Hygiene Plan SOP 90_0001 in conjunction with the Safety orientation

3.2 All sample and material handling should be done in a hood while using proper protective equipment to minimize exposure to liquid or vapor. Minimum personnel protective equipment includes the use of laboratory safety glasses, a lab coat or apron, and protective gloves.

3.3 The MSDSs for the concentrated chemicals used in the laboratory are kept on file in a central location that is available for all employees to review.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

4.1 Sample preservation for soil samples is discussed in SOP 20_5035. Aqueous samples are preserved with 1:1 HCl at a ratio of 100 µL to 40 ml.

4.2 Samples for analysis using this SOP must be stored in tightly sealed vials with Teflon-lined silicone septum seals in which the original sample was collected. All samples are stored in a refrigerator at 4 ± 2°C within the laboratory. The soil samples are stored in a separate refrigerator from the aqueous samples in the VOC laboratory. Samples for volatile analysis must be analyzed within 14 days of sample collection

4.3 Samples known to contain high concentrations of VOCs are stored in a separate refrigerator located in the sample storage room. This is to prevent cross contamination.

4.4 Trip blanks for aqueous samples are prepared with organic free water. The water comes from a carbon filtration system located in the VOA lab and is periodically checked for low level VOCs.

4.5 A trip blank should accompany each batch of 20 or less VOC samples. This trip blank must accompany the associated samples at all times.

4.6 Refrigerator blanks are prepared like trip blanks and stored in the refrigerator with samples. These blanks are analyzed weekly to check for possible cross contamination from samples. To prevent cross contamination from sample to sample, all sample containers must be airtight. If the blank is greater than the MRL, then notify operations manager immediately.

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5.0 INTERFERENCES AND POTENTIAL PROBLEMS

5.1 Major sources of contamination are volatile materials in the laboratory and impurities in the inert purging gas and in the sorbent trap. The use of non-polytetrafluoroethylene (PTFE) thread sealant, plastic tubing, or flow controllers with rubber components should be avoided since such materials out-gas organic compounds which will be concentrated in the trap during the purge operation.

5.2 Analyses of reagent blanks provide information about the presence of contaminants. When potential interfering peaks are noted in blanks, the analyst should change the purge gas source. Subtracting blank values from sample results is not permitted.

5.3 Interfering contamination may occur when a sample containing low concentrations of volatile organic compounds is analyzed immediately after a sample containing high concentrations of volatile organic compounds. After analysis of a sample containing high concentrations of volatile organic compounds, one or more instrument blanks must be analyzed to check for cross contamination.

5.3.1 This interference may be prevented by rinsing the purging apparatus and sample syringes with portions of organic-free reagent water between samples.

5.3.2 In extreme situations, the whole purge and trap device may require dismantling and cleaning.

5.4 Special precautions must be taken to avoid contamination when analyzing for methylene chloride. The analytical and sample storage area must be isolated from all atmospheric sources of methylene chloride. Otherwise, random background levels will result. Since methylene chloride will permeate through PTFE tubing, all gas chromatography carrier gas lines and purge gas plumbing must be constructed from stainless steel or copper tubing. Laboratory clothing worn by the analyst should be clean since clothing previously exposed to methylene chloride fumes during liquid/liquid extraction procedures can contribute to sample contamination.

5.5 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal into the sample during shipment and storage. A trip blank prepared from organic-free reagent water and carried through the sampling and handling protocol can serve as a check on such contamination.

5.6 This procedure can be used to quantitate most volatile organic compounds that have boiling points below 200°C and that are insoluble or slightly soluble in water. Volatile water-soluble compounds can be included in this analytical technique. However, for the more soluble compounds, quantitation limits are approximately ten times higher because of poor purging efficiency. Such compounds include low-molecular-weight-halogenated hydrocarbons, aromatics, ketones, nitriles, acetates, acrylates, ethers and sulfides.

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6.0 EQUIPMENT/APPARATUS

6.1 **Microsyringes**: 10 µL, 25 µL, 50 µL, 100 µL, 250 µL, 500 µL, and 1,000 µL. These syringes should be equipped with a 20 gauge (0.006" ID) needle.

6.2 **Disposable micropipettes**: 10, 50, 100 and 200 µL volumes. Purchased from Drummond.

6.3 **Syringe**: 5 ml and 25 ml glass, gas-tight, with Luerlock tip.

6.4 **Balance**: Top-loading balance capable of weighing 0.1 g.

6.5 **Micro-reaction vessels**: 1.0 ml and 5.0 ml purchased from Supelco. Catalogue No. 3-3293 with Mininert Caps. Clean reaction vessels by placing in oven at 105°C overnight.

6.6 **Volumetric flasks**: of various sizes with ground glass stoppers –Class A.

6.7 **Vials**: 40 ml, with pierceable Teflon screw cap top.

6.8 **Spatulas**: Stainless steel.

6.9 **Disposable pipettes**: Pasteur.

6.10 **Culture tubes**: 10 x 150 mm. Kimball No. 45060-19150. Culture tubes are only used once, then discarded.

6.11 **Syringe Valve**: Two-way with Luerlok ends.

6.12 **Chamber heaters**: Tekmar sample heater, capable of maintaining the purging chamber to within 1°C over the temperature range of ambient to 100°C.

6.13 **Purge and trap devices**: (A) Tekmar 3100 Purge and Trap Concentrator. (B) Archon Purge and Trap Auto-Sampler System (Varian)

6.13.1 The Archon Purge and Trap Auto-sampler is designed to Automate the tedious sample handling procedures associated with purge and trap analyses for volatile organic compounds (VOCs) under current EPA methods. The Archon can be used for drinking water, wastewater, soil and solid analyses.

6.13.2 **Function Keys**:

A) Method: Methods are entered using a keypad with LCD screen. All information can be edited using keypad. ***NOTE: Settings are variable and may change depending on analysis requirements.***

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Method parameters consist of:

	MS1/MS3/MS4
Sample Type (water/soil)	water
First Vial(1-51)	variable

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Last Vial(1-51)	variable
Sample Volume(0-25ml)	05
Dilution Factor	variable
Rinse Volume(0-25ml)	07
# Rinses(0-20)	01
Standard 1(yes/no)	variable
Standard 2(yes/no)	variable
S. Preheat Stir(yes/no)	no
Stir(yes/no)	no
W. Stir Time(0-9.9min)	0.0
W. Settle Time(0-99sec)	0.0
Syringe Flush(0-20)	01
Preheat(yes/no)	no
Preheat Temp(amb-140C)	--
Preheat Time(0-99.9min)	--
Purge Time(0-999.9min)	--
Desorb Time(0-99.9min)	1.0
Operation Mode(Local/Remote)	Remote
Cycle Timer(0-99.9min)	0.0
Aux Timer(0-999min)	0.0
Link to Method	variable

B) **Auto**: Starts purge sequence. Initiates single blank runs. Single blank runs can be programmed after specific sample vials within a particular method. These method blanks allow the cleaning of not only the Archon, but also the purge and trap and GC. The blank water is withdrawn from the blank water reservoir, which is pressurized by helium gas at 20-25 psi. Before changing the blank reservoir, the "Blank Water Reservoir and Standard Helium" switch must turned off. This switch is located on the right rear panel inside the Archon. After refilling the reservoir, this switch must be turned on.

C) **Manual**: allows for manual injection of samples.

D) **Flush**: allows flushing of Archon components using water from Blank Water Reservoir

E) **System**: provides access to all settings, maintenance, calibration, and options of the Archon.

F) **Pause/Stop**: allows analyst to pause or abort sequence.

6.13.3 The sample is purged in a 5ml fitted purge tube. The purge gas passes through the water column as finely divided bubbles, each with a diameter of less than 3 mm at the origin. The purge gas is introduced no more than 5 mm from the base of the water column.

6.13.4 The trap is at least 25 cm long and has an inside diameter of at least 0.105". This trap is commercially available from Supelco (Vocarb 3000, Catalogue

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No. 2-4920).

6.13.5 The desorber is capable of rapidly heating the trap to 225°C and performing the bake out step at 260°C.

6.13.6 The chamber heater is capable of maintaining the purge device at 40°C ±1°C.

6.14 **Gas chromatograph**: HP5890 Series II or HP-6890. A complete analytical system with a temperature programmable gas chromatograph suitable for on-column injection and all required accessories including syringes, analytical columns and gases.

6.15 **Column**: (A). 75 M x 0.53 mm ID or (B). 30M x 0.25 mm ID, DB624 commercially available from J&W Scientific (or equivalent).

6.16 **Mass spectrometer**: HP5971MSD or HP5972MSD - Capable of scanning from 35 to 260 AMU every 1 second or less, utilizing 70 volts electron energy in the electron impact ionization mode and producing a mass spectrum which meets all the criteria listed in section 11.1 when 50 ng of 4-Bromofluorobenzene (BFB) is injected through the gas chromatograph inlet.

6.17 **Data system**:

6.17.1 **Computers:** The Volatiles laboratory has three GC/MS systems analyzing method 5030B/8260B. VOA MS1 has an AST computer with a Windows 95 operating system. VOA MS3 and MS4 have DELL computers with Windows NT operating system. All computer systems are networked to a Windows 2000 server, which is the destination of all files. A differential back-up is performed nightly and a full back is performed each weekend using Veritas Backup Exec to tapes. As the systems acquires and stores data onto the server, the server becomes full. The data is downloaded and archived onto CDs.

6.17.2 **Software:** HP Enviroquant MSChemstation - The software is interfaced to the mass spectrometer detector and allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer software allows searching of any GC/MS data file for ions of a specified mass and plotting such ion abundance versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). The software is also capable of integrating the abundance in any EICP between specified time or scan number limits. The most current version of the EPA/NIST Mass Spectral Library is also available. Current versions VOA MS1:G1032C version C.01.00, VOA MS3:G1701BA Version B.01.00, and VOA MS4: G1701AA version A.03.00.

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7.0 REAGENTS AND STANDARDS

7.1 Reagents:

7.1.1 **Methanol**: Purge and Trap grade, purchased from Fisher, Catalog No.A453-500.

7.1.2 **Reagent Water**: Water in which interference is not observed at ½ the method reporting limit (MRL) of the parameters of interest. DI water obtained from the VOA laboratory fits the criterion.

7.2 Standards:

7.2.1 **Primary Standards**: obtained from a commercial source. They are stored in the freezer at -10°C to -20°C. After opening; they are stored in a vial with a mininert valve. All Certificates of Analysis (COAs) are marked with the appropriate Primary Standard ID and placed in a logbook. Primary standards are not to be used after the manufacturer's expiration date. Once a standard has been opened, it may not be used after 6 months. A copy of the COAs, which details the compounds found in the mixes, are in Attachment A.

Manufacturer	Standard	Catalog Number	Conc. (µg/ml)
Restek	Ketones	30006	5000
Restek	Acrolein	30645	5000
Restek	Oxygenates	30465	2000/10,000 (TBA)
Restek	Custom	558360	2000
Restek	Gases	30042-510	2000
Restek	Cal Mix#1	30633	2000
Restek	2-CEVE	30265	2000
Restek	Vinyl Acetate	30216	2000
Supelco	1,4-Dioxane	44-2251	10,000*
Ultra	Surrogate	STM-530-1	2500
Accustandard	1,3,5-Trichlorobenzene	As-E0176	5000

*The primary standard for 1,4-Dioxane is made up from neat by diluting 10 µL of neat 1,4-Dioxane into a 1.0 ml volumetric flask

7.2.2 **Working Calibration Standard (25 µg/ml)**: Prepared standards are stored in micro reaction vessels or vials capped with mininert valve. Standards are checked frequently for signs of degradation or evaporation. Working calibration standards are used for preparing calibration curves and daily continuing calibrations.

7.2.2.1 Into 5ml volumetric flask add the following: 125 µL of Ketone std., 62.5 µL each of gas, Custom std., Cal Mix#1, Vinyl acetate, and oxygenate standards, 312.5 µL of 2-CEVE std, 25µL of Acrolein std, 25µL of 1,3,5-Trichlorobenzene, 237.5 µL 1,4-Dioxane and 50 µL of surrogate standard. Volumize to 5ml with MeOH. Final concentration equals 125 µg/ml for the Ketones and 2-CEVE, 500 µg/ml of 1,4-Dioxane, and 25 µg/ml for all other analytes including

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surrogates.

7.2.3 **Secondary Source/BS/Matrix spike standards**: obtained from a commercial source. They are stored in the freezer at -10°C to -20°C. After opening; they are stored in a vial with a mininert valve. All Certificates of Analysis must be kept on file.

Manufacturer	Standard	Catalog Number	Conc. (µg/ml)
Accustandard	TCL Ketone Mix	CLP-022K-25x	5000
Ultra	Acrolein	RCC-150	10,000*
Accustandard	Method 502.2 VOC	M-502-10x	2000
Ultra	VOC Mix	DWm-592-1	2000
Ultra	Custom Std	CUS-10604	2000
Accustandard	2-CEVE	M-601C-10x	2000
Supelco	Vinyl acetate	4-0327	5000
Ultra	1,4-Dioxane	RCC-180	10,000*
Accustandard	tert-Butyl Alcohol	S-410	2000

*The primary standard for 1,4-Dioxane and Acrolein is made from neat product by diluting 10 µL into a 1.0 ml volumetric flask.

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7.2.3.1 **Initial Calibration Verification- ICV/BS/BSD/Matrix spike standard (25 µg/ml):** Used to check the primary standard. Into 5ml volumetric flask containing approximately 3 ml of MeOH add 62.5 µl each of Custom std., VOC mix , 502.2 mix and 25 µL of Vinyl acetate. Add 12.5 µL of Acrolein, 250 µL of 1,4-Dioxane, 100 µL TCL Ketone Mix, and 312.5 µL tert-Butyl Alcohol and 2-CEVE. Volumize to 5ml with MeOH. Individual components will have a final concentration of 25 µg/ml with the exception of 1,4-Dioxane at 500µg/ml. A copy of the COAs, which details the compounds in the mixes, is attached.

7.2.4 **Surrogate Standards**: The surrogate solution is made up of Dibromofluoromethane, Toluene-d_8, 4- Bromofluorobenzene, and 1,2-Dichloroethane-d_4. The mixed stock surrogate solution is purchased from Ultra at 2500 µg/ml (cat no. STM-530-1). The stock solution is logged into the primary standard logbook upon receipt. Several surrogate standard solutions are prepared from the stock at different concentrations in methanol.

7.2.4.1 **Surrogate mix**: For soil/sediment samples, the preparation of this solution is discussed in SOP# 20_5035.

7.2.4.2 **Surrogate/Internal standard QC mix MS1, MS3, and MS4**: To a 5 ml volumetric flask containing approximately 3 ml of methanol add 250 µL of stock surrogate and stock internal standard. Volumize with methanol to give a final concentration of 125 µg/ml. Transfer to reservoir #1 on the Archon. Addition of 1uL of this standard by the Archon to 5.0 ml of sample is equivalent to 25 ppb.

7.2.5 **Internal Standards**: The internal standards are Pentafluorobenzene,

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Chlorobenzene-d_5, and 1,4-Dichlorobenzene-d_4. The mixed internal standard solution is purchased from Restek at 2,500 µg/ml (Cat No. 30074). See SOP 20_5035 for internal standard solution for soil calibration.

7.2.5.1 **Internal Standard for calibration:** Prepare by addition of 50 uL of Internal Standard solution into a final volume of 5 ml MeOH, final concentration is 25 ug/ml.

7.2.6 **Continuing Calibration Verification Standard (CCV):** MS1, MS3, and MS4: Addition of 50 uL of the working calibration standard (See 7.2.2.1) to 50.0 ml of organic free water is equivalent to 25 ppb. This solution is prepared fresh daily.

7.2.7 **Blank Spike/Blank spike duplicates (BS/BSD)**: Prepared by adding 40 uL of Blank spike solution (7.2.3.1) to 100 ml DI water in a 100 ml volumetric flask for a final concentration of 10 ug/L. Blank spike concentrations are to be at the midpoint (median standard level) of the calibration curve.

7.2.8 **4-Bromofluorobenzene (BFB) Standard**: A stock solution in methanol is purchased from Supelco at a concentration of 2,000 µg/ml (cat. no. 4-8083).

7.2.8.1 Add 62.5µL of the stock solution to a 5.0-ml volumetric flask and dilute with methanol to produce a 25-µg/ml solution. A 25 ug/L standard is purged to check the instrument tune.

7.2.9 **Matrix Spiking Standard**: prepared as in section 7.2.3.1

7.2.9.1 **Working Matrix Spike Solution**: Prepare aqueous matrix spikes by adding 40 uL of matrix spike solution to 100 ml sample in a 100 ml volumetric flask for a final concentration of 10 ug/L.

8.0 PROCEDURE

8.1 Initial calibration for purge-and-trap procedures:

8.1.1 ESS Laboratory's policy is that the audit trail on the Chemstation/Enviroquant software is always on. This ensures that any changes made to the instrument operating method be documented through the audit trail.

8.1.2 GC/MS operating conditions: The following parameters are setup through Hewlett Packard MS EnviroQuant. A copy of an EnviroQuant method is in Attachment B.

Electron energy:	70 volts		
Mass range:	35 amu - 260 amu		
Scan time:	1.5 sec/scan		
Initial column temp..:	40°C		
Initial column hold time:	4.0 min.		
Column temp. program:	Rate	Final Temp	Final Time

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Level 1	8°C/Min	150°C	2 min
Level 2	35°C/Min	220°C	0-4.25 min
Injector temperature:	200°C		
Transfer line temperature:	280°C		
Carrier gas (Helium):	15 cm^3/ml		

8.1.3 Assemble a purge-and-trap device that meets the specification in Section 6.1.3. New traps are conditioned one hour at 270°C in the bake mode. Prior to daily use bake trap for 8 minutes at 260°C

8.1.4 Just prior to any analysis of samples or standards, the run logbook should be completely filled out (See Attachment C). Each instrument has its own run logbook. Any unusual observations are included in the comment section of the logbook. The information that must be recorded is:

8.1.4.1 **Batch Date**: Date analytical sequence set-up

8.1.4.2 **Vial #**: Position the standard/sample vial is placed in the auto-sampler

8.1.4.3 **File #**: File ID assigned to standard/sample in sequence

8.1.4.4 **Lab ID**: The ID of the sample or standard. Sample IDs are assigned at log-in by ELEMENT (ex: 0508001) and Batch QC samples contain the batch ID (ex BG50801-blk1, BG50801-bs1, BG50801-bsd1, …)

8.1.4.5 **Method**: Record the method used. The method includes instrument operating parameters and initial calibration for quantitation.

8.1.4.6 **Comments/Dilution/Standard ID**: Record comments and unusual observations. If a sample is run at a dilution, then record the dilution information in the comments section. (Ex. 25x dilution on aqueous sample: 0.2/5 ml) Record standard/spike ID for Tune, CCV, blank spike/blank spike duplicates, and matrix spike/matrix spike duplicates.

8.1.4.7 **Analyst**: Analyst must record their initials.

8.1.5 A tune check must be performed before the initial calibration curve. This is accomplished by purging a 25 ug/L standard of the 4-Bromofluorobenzene standard (See section 7.2.8) onto the GC/MS column. Analysis is not to begin until the 4-BFB spectra meets the criteria in Section 11.1. Evaluation of the ion abundance is performed by using one of the following scan scenarios:

- Use one scan at the peak apex,
- Use the one scan either directly preceding or following the apex,
- Use the mean of the apex and the scans preceding and following the scan, or
- Use the average across the entire peak.

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8.1.5.1 The analysis of all calibration standards must be accomplished within 12 hours of this tune check.

8.1.6 Prepare the standards for generating the calibration curve using the Working Standards (see section 7.2.2) and Internal Standard solution (see Section 7.2.4.1) as follows:

8.1.6.1 **Soil Method:** The calibration curve for soil/sediment samples is discussed in SOP 20_5035.

8.1.6.2 **Water Method for MS1, MS3, and MS4:** seven standards (0.4, 1.0, 5.0, 10, 25, 50, and 100 ppb) are run. The 5 ppb is prepared in a 50ml volumetric by adding 10µL of 25 ug/ml working calibration standard (7.2.2) to 50 ml of DI water. 50 ul of internal standard mix (7.2.5.1) is then added for a final concentration of 25 ppb. The flask is inverted three times and the solution is transferred to a 40 ml VOA vial leaving no headspace. The 0.4, 1.0, 10, 25, 50, and 100ppb standards are prepared by adding 0.8, 2.0, 20, 50, 100, and 200µL of working standard as described above, the amount of internal standard added remains constant for all the standards. These are then placed in the Archon for purge and trap analysis.

8.1.7 Carry out the purge-and-trap analysis procedure as described in Section 8.3.

8.1.8 EnviroQuant will tabulate the area response of the characteristic ions (see Table 2) against concentration for each compound and internal standard. It will also calculate response factors (RF) for each compound relative to its internal standards according to the calculations in Section 9.1.

NOTE: HP EnviroQuant software calculates and reports response factors for the analyst. It is each analyst's responsibility to become familiar with the software by reading the available manuals. **After generating the initial calibration curve in Enviroquant, the analyst must visually check that each calibration standard was entered into the new calibration method. This is accomplished by checking that the area response for one compound from each calibration standard's printout corresponds to the area account listed in the calibration method in Enviroquant.** All of the analytes along with their retention times, retention windows, and the ions used for qualitative analysis are listed in Attachment B.

8.1.9 The average RF is calculated and recorded in the EnviroQuant Method for each compound. A system performance check is made before this calibration curve is used. Five compounds (the System Performance Check Compounds or SPCCs) are checked for a minimum average response factor. These compounds are Chloromethane, 1,1-Dichloroethane, Bromoform, 1,1,2,2-Tetrachloroethane and Chlorobenzene. The minimum acceptable RFs are 0.1 for Chloromethane, 1,1-Dichloroethane, and Bromoform, and 0.3 for Chlorobenzene and 1,1,2,2-Tetrachloroethane. These compounds are used to check compound instability and to check for degradation of the

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system, such as active sites in transfer lines or trap failure. ***NOTE: The minimum response factor for all other compound should not be less than 0.01. ESS Laboratory has found that the following poor performing compounds do not meet this criterion: Acetone, 2-Butanone (MEK), and 1,4-Dioxane.***

8.1.10 Using the RFs from the initial calibration, EnviroQuant software calculates the percent relative standard deviation (%RSD) for all compounds using the equation in Section 9.2. The %RSD should be less than 15% for each compound, except for the following continuing calibration compounds (CCCs) 1,1-Dichloroethene, Chloroform, 1,2-Dichloropropane, Toluene, Ethylbenzene and Vinyl Chloride, where the %RSD must be less than 30%.

8.1.10.1 Linearity - If the %RSD of any compound is 15% or less, then the relative response factor is assumed to be constant over the calibration range, and the average relative response factor may be used for quantitation.

8.1.10.2 When the RSD exceeds 15%, the plotting and/or visual inspection of the calibration data can be a useful diagnostic tool. The inspection may indicate analytical problems, including errors in standard preparation, the presence of active sites in the chromatographic system, analytes that exhibit poor chromatographic behavior, etc.

8.1.10.3 If a %RSD > 30 percent is measured for any method analyte (50% for poor purgers), then corrective action to eliminate a system leak and/or column reactive sites is required before re-attempting calibration. Poor purging compounds are listed in Table 2.

8.1.10.4 If the RSD of **any** compound exceeds 15% (never to exceed 30% except for poor purgers), then one of the following options must be applied to the GC/MS initial calibration in this situation or a new initial calibration must be performed.

8.1.10.4.1 Adjust the instrument and/or perform instrument maintenance until the RSD of the calibration meets the 15% QC limit. This option would apply in those instances where a linear instrument response is expected.

8.1.10.4.2 Narrow the calibration range until the response is linear. If the low standard is below the estimated quantitation limit (i.e., for the poor purgers in a commercially available prepared standard mix), then this standard may be dropped. Recalculate the RSD without this standard to see if the RSD meets the QC limit. It would be recommended that a new standard be prepared at a concentration between the existing fourth and fifth calibration standards, analyzed, and a new RSD calculated with all five points.

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8.1.10.4.3 Use a linear calibration. This would be achieved by performing a linear regression of the instrument response versus the concentration of the standards, **do not** force the curve through zero. In order to be used for quantitative purposes, the Correlation Coefficient r, must be greater than or equal to 0.995. This can easily be accomplished within the EnviroQuant software (see attachment B).

8.1.10.4.4 Use a non-linear calibration model. This option should be used only after exhausting the other three options, or in situations where the analyst knows that the instrument response does not follow a linear model over a sufficiently wide working range. (Coefficient of Determination, COD > 0.99).
When non-linear calibration is employed a minimum of six calibration points must be used for second order and seven calibration points for third order.

8.1.11 Immediately after the initial calibration has been generated then the initial calibration has to be verified using a second source standard. The percent recovery for the independent calibration verification standard is 80-120% for all CCC compounds and 70-130% for all other compounds. ***When samples are to be to be run for the DoD , all compounds must have ≤20% difference with no allowance.***

8.1.12 The RRT's are established from the 25 ppb mid-point standard in the initial calibration

8.2 Daily GC/MS analysis: Just prior to all samples and standard analysis, the run logs are filled out according to 8.1.4.

8.2.1 Prior to the analysis of samples, purge a 25 ug/L standard of 4-BFB (50 uL of standard is added into 50ml DI water, Section 7.2.8). The resultant mass spectra for BFB must meet all of the criteria in Section 11.1 before sample analysis begins. This criteria must be met each 12-hour shift.

8.2.2 The initial calibration curve must be verified each 12-hour shift. This is accomplished by analyzing a calibration standard (7.2.7) at 25 µg/L (MS1, MS3, and MS4). Determine if the SPCCs meet criteria from section 8.1.9. Determine if the CCCs listed in section 8.1.10 have % Deviation less than 20% with all other analytes having a % Deviation less than 30%, poor purging compounds (Ketones, 2-CEVE) must be less than 50%. Compounds not analyzed for in the batch of samples do not need to meet the criteria. Continuing calibrations used for 8021 analysis must have <20% RSD. ***When samples are to be to be run for the DOD, all compounds and surrogates should have <20% Drift/Deviation, see Section 11.4 for expanded criteria.*** For compounds analyzed for Method 624, continuing calibration criteria are met or exceeded by 8260 criteria. **Refer to**

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Attachment E for Mass DEP MCP requirements.

8.2.3 The internal standard responses and retention times in the continuing calibration standard should be evaluated immediately after data acquisition. If the area for any of the internal standards changes by a factor of two (-50% to +100%) or the retention time changes by ± 0.5 minutes from the **mid-point standard of the initial calibration**, the mass spectrometer should be checked for defects. The relative retention time of each compound in each calibration run must agree to within 0.06 Relative Retention Time (RRT) Units. The RRT's in the continuing calibration standard is compared to the RRT's from the 25 ppb standard in the ICAL.Analysis is not to continue until problem has been resolved.

8.2.4 Method blank analysis should be performed at the following frequency:

8.2.4.1 For the analysis of volatile compounds, a method blank analysis must be performed once for each 12-hour shift and must be analyzed before any samples.

8.2.4.2 A method blank for volatile analysis should contain less than or equal to the Method Reporting limit of methylene chloride. For all other TCL compounds, the method blank should contain less than ½ MRL of any single TCL analyte. See section 11.5 for instructions on corrective action when criteria are exceeded.

8.2.5 Blank Spike/Blank spike duplicate analysis must be performed once for each 12-hour shift, after the tune and CCV, and must be analyzed before any samples. BS/BSD concentrations are to be at the midpoint (median standard level) of the calibration curve. See section 10.0 for BS/BSD acceptance criteria.

8.2.5.1 See 7.2.7 for BS/BSD preparation.

8.3 GC/MS analysis: All samples and standards are analyzed under the same conditions in the associated initial calibration. Run logs are filled according to section 8.1.4.

8.3.1 Aqueous samples

8.3.1.1 All samples and working standard solutions should be allowed to warm to ambient temperature before analysis.

8.3.1.2 Set up the GC/MS system as outlined in Section 8.1.1.

8.3.1.3 BFB tuning criteria and daily GC/MS calibration criteria must be met (Section 11.1) before analyzing samples.

8.3.1.4 Sample or standard vials are placed in the Archon. The Archon will transfer 5 ml of sample/standard to a 5ml fitted purge tube (MS1, MS3, and MS4). The process of taking an aliquot destroys the validity of remaining volume of an aqueous sample for future

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analysis.

8.3.1.5 The Archon is programmed to add 1µL of Surrogate/Internal Standard (Section 7.2.4.2) to the sample. The Surrogate/Internal Standard QC Mix is to be added to each blank, sample and spiked sample.

8.3.1.6 Purge the sample for 11.0 ± 0.1 min at ambient temperature.

8.3.1.7 At the conclusion of the purge time, the purge-and-trap will sound a ready signal from the GC and begin to desorb the trap while initiating the chromatographic temperature program and the CHEMSTATION data acquisition.

8.3.1.8 Before reusing any chamber, it must be rinsed with three-5 ml aliquots of reagent water.

8.3.1.9 After the sample has been desorbed for one minute, the LSC 2000 will recondition the trap by heating to 260°C while back flushing with Helium. This step will take 10 minutes.

8.3.1.10 When necessary to composite samples for analysis, the following procedure is used:

8.3.1.10.1 Add 5 ml of each sample (up to 5 samples are allowed) to a 25 ml glass syringe. Special precautions must be made to maintain zero headspace in the syringe. Larger volumes of a smaller number of samples may be used, if equal volumes of each sample are composited.

8.3.1.10.2 The samples must be cooled to 4°C or less during this step to minimize volatilization losses. Sample vials may be placed in a tray of ice during the processing.

8.3.1.10.3 Mix each vial well and draw out a 5-ml aliquot with the 25-ml syringe.

8.3.1.10.4 Once all the aliquots have been combined in the syringe, invert the syringe several times to mix the aliquots. Introduce the composited sample into the instrument, using the method of choice.

8.3.1.10.5 If less than five samples are used for compositing, a proportionately smaller syringe may be used, unless a 25-ml sample is to be purged.

8.3.1.11 If a sample or a dilution of the sample has a concentration of analytes that exceeds the initial calibration range, the sample must be reanalyzed at a higher dilution. The ARCHON can be set to dilute up to 100x. Dilutions can also be performed directly in 5-ml

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syringe by adding an aliquot of sample to reagent water using a micropipette. The follow procedure is used to dilute aqueous samples. All steps must be performed without delay, until the diluted sample is in a gas-tight syringe.

8.3.1.11.1 Dilutions may be made in volumetric flasks (10-100 ml). Select the volumetric flask that will allow for the necessary dilution. Intermediate dilution steps may be necessary for extremely large dilutions.

8.3.1.11.2 Calculate the approximate volume of organic-free reagent water to be added to the volumetric flask, and add slightly less than this quantity of organic-free reagent water to the flask.

8.3.1.11.3 Inject the appropriate volume of the original sample from the syringe into the flask. Aliquots of less than 1 ml are not recommended. Dilute the sample to the mark with organic-free reagent water. Cap the flask, invert, and shake three times. Repeat above procedure for additional dilutions.

8.3.1.11.4 Dilutions are documented in the analytical run logbook.

8.3.1.12 When a sample is analyzed that has any target compound at a level exceeding twice the initial calibration range, this analysis should be followed by a blank reagent water analysis in the purge chamber to demonstrate that system is clean. If the blank analysis is not free of interferences, the system should be decontaminated. Sample analysis may not resume until a blank can be analyzed that is free of interferences.

NOTE: Since the analytical system is automated the analyst may not be aware that a high concentration sample has been analyzed. If analysis of subsequent samples occurs, then the presence of the high concentration analyte makes re-analysis of that sample necessary to determine if carryover has occurred.

8.3.1.13 For matrix spike analysis, in a 100 ml volumetric flask add 40 µL of the matrix spike solution (Section 7.2.3.1) to the 100 ml of sample. Invert three times and pour into 2 VOA vials.

8.3.1.14 All dilutions should keep the response of the major constituents in the upper half of the linear range of the curve.

8.3.2 Dilutions for soil/sediment samples are discussed in SOP 20_5035.

8.3.2.1 The procedure for running soil/sediment samples is discussed in SOP 20_5035.

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8.4 Data interpretation

8.4.1 Qualitative identification

8.4.1.1 An analyte (e.g. those listed in Table 2) is identified by comparison of the sample mass spectrum with the mass spectrum of a standard of the compound obtained on the user's GC/MS. These standard reference spectra are obtained through analysis of the calibration standards. To compare the sample and reference spectra, go into the EnviroQuant software (QEDIT). The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity - any ions over 30% if less than three such ions are present. The analytes are listed in Attachment B for each analyte.

8.4.1.2 The requirements for qualitative verification by comparison of mass spectra are:

8.4.1.2.1 The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine, where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time, will be accepted as meeting this criterion.

8.4.1.2.2 The RRT of the sample component is compared to the RRT from the 25 ppb standard in the ICAL and must be within ±0.06 RRT units of the RRT of the standard component.

8.4.1.2.3 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%).

8.4.1.2.4 Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between the two isomer peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs. ESS Laboratory currently reports m- and p-xylenes together due to inability of resolving the isomers.

8.4.1.2.5 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with

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shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra are important. Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra, and in qualitative identification of compounds. When analytes co-elute (i.e., only one chromatographic peak is apparent), the identification criteria can be met, but each analyte spectrum will contain extraneous ions contributed by the co-eluting compound.

8.4.1.2.6 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the type of analyses being conducted. When serving the role of QA (or referee) laboratory, tentatively identified compounds (TICs) shall always be reported. Guidelines for making tentative identification are listed in section 11.9.3.

8.4.2 Quantitative Analysis

8.4.2.1 Compounds, which have been identified, are quantified by the internal standard method, utilizing the integrated abundance of the primary ion. The internal standard used shall be those listed in Section 8.1.7. The EnviroQuant method is setup to calculate the concentration of the analytes of interest in ng/ml using the initial calibration results stored in the method (Attachment B). This raw data number does not account for deviations in sample volume, sample weight, dilution factors, and % moisture. To include these other factors, see section 9.0.

9.0 CALCULATIONS

9.1 Response Factor (RF)
RF=(As x Cis)/(Ais x Cs)

Where:

A_S = Peak area (or height) of the analyte or surrogate.

A_{IS} = Peak area (or height) of the internal standard.

C_S = Concentration of the analyte or surrogate, in µg/L.

C_{IS} = Concentration of the internal standard, in µg/L.

9.2 Percent Relative Standard Deviation (%RSD)
$\%\ RSD = (SD/\ RF_{Average}) \times 100\%$

Where:

RSD = Relative standard deviation.

RF = Mean of 5 initial RFs for a compound.

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SD = Standard deviation of average RFs for a compound.

9.3 Percent Difference

$\%Difference = ((RF_{Average} - RFc)/ RF\ Average) \times 100\%$

Where:

$RF_{Average}$ = Average response factor from initial calibration

RF_c = Response factor from current verification check standard.

9.4 Percent Drift

$\%Drift = ((C1-C0)/ C1) \times 100\%$

Where:

C_1 = compound standard concentration

C_0 = measured concentration using selected quantitation method

9.5 Concentration of Target Analytes in Water and Water-Miscible Waste Concentration

$\mu g/L = (Ax)(Cs)(D) / (Ais)(RF_{Average})(Vs)$

Where:

A_X = Area (or height) of the peak for the analyte in the sample.

A_{is} = Area (or height) of the peak for the internal standard.

C_S = Mass (amount) of the internal standard in the concentrated sample extract (ng). This is not just the mass injected into the instrument, but the total mass of internal standard in the concentrated extract.

D = Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution was made, D = 1. The dilution factor is always unitless.

$RF_{Average}$ = Mean response factor from the initial calibration.

V_S = Volume of the aqueous sample extracted or purged (ml). If units of liters are used for this term, multiply the results by 1000.

Using the units specified here for these terms will result in a concentration in units of ng/ml, which is equivalent to µg/L.

9.6 Concentration of Target Analytes in Sediment/Soil, Sludge, and Waste – see SOP 20_5035.

10.0 QUALITY ASSURANCE/QUALITY CONTROL

Accuracy and Precision

Laboratory personnel must demonstrate initial proficiency for each sample preparation method/matrix that he or she performs. New employees must successfully demonstrate initial proficiency prior to independently performing analysis on real samples. This is accomplished by generating data of acceptable accuracy and precision for target analytes in

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a clean matrix. Initial proficiency results will become part of each employee's training file.

QC Sample Preparation:
Spiking Solution: Four QC samples must be prepared from a spiking solution with the analytes of interest. The spiking solution must be made using standards **prepared independently from those used for calibration.** The samples must be prepared at a concentration that would result in data falling within the middle of the calibration curve. In most cases the blank spike or matrix spike solution is used. Prep: The samples are prepared in a clean matrix. In most cases this initial demonstration will simply be a matter of preparing four blank spikes with a batch of samples.

Sample Analysis:
The four QC samples must be analyzed within the criteria of the method being evaluated. The QC samples must be handled in exactly the same manner as actual samples.

Accuracy Calculation:
Accuracy is defined as the closeness of agreement between an observed value and an accepted reference value. Each of the four spiked samples will be calculated for percent recovery. The average of the percent recovery values is the accuracy result.

Precision Calculation:
Precision is defined as the agreement of a set of replicate measurements without assumption of knowledge of the true value. Precision is estimated by the relative standard deviation (RSD) of the four QC samples.

$$\%RSD = (s / \bar{x})\ 100\ \%$$

Where:
s = Standard Deviation of a finite number of values. On a scientific calculator use the **σxn-1** key.

$\bar{x}$ = The average of the four QC sample % recoveries.

Reporting Accuracy and Precision
Accuracy and Precision data should be presented with the following minimum info:

Matrix: Prep Method:
Analysis Method: Date Analyzed:
Accuracy: Precision:

Parameter	*% Rec. QC 1*	*% Rec. QC 2*	*% Rec. QC 3*	*% Rec. QC 4*	*Average Recovery*	*Standard Deviation*	*%RSD*

Interpretation of Results: Percent recovery is to be within 80-120% for all analytes with a RPD of ≤20%. For the full 8260B list a maximum of 5 sporadic marginal failures are allowed with the expanded criteria of 60-140%.

10.1 A preparation batch is defined as a group of up to 20 field samples of similar matrix

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that have been prepared at the same time or time sequence with the same lots of reagents for the same analysis. In addition to the samples, each prep batch will contain at a minimum, a method blank, a blank spike, blank spike duplicate, a matrix spike, and a matrix spike duplicate. An analytical/instrument batch is defined as samples that are analyzed together within the same time period or in continuous sequential time periods. The analytical batch includes QC requirements as defined by the analytical (determinative) method. For example, each injection sequence would begin with a tune and CCV (or tune, calibration and ICV), followed by a method blank, BS/BSD, MS/MSD, etc. and remaining field samples. Prep batches may be continuously strung together in these run sequences, as long as the analytical batch QC requirements meet the acceptance criteria established within the appropriate SOP. If all field and QC samples could not be completed within the 12-hour clock, then another tune, CCV, and MB would be required. Each analytical sequence must be documented using the run log in Figure 1.

10.2 Perform BFB tune every 12 hours. Tuning acceptance criteria are presented in Section 11.1. The computer software will evaluate the tune information. The analyst should be aware of the process used.

10.3 Run a 7-point initial calibration curve, using the primary source standards each time major instrument maintenance occurs, or if the CCV does not meet acceptance criteria. Acceptance criteria are presented in Section 11.2.

10.4 Run initial calibration verification (ICV) standard using secondary standards (7.2.3) following the initial calibration curve. Acceptance criteria are listed in Section 11.3.

10.5 Run a mid-point Continuing Calibration Verification (CCV) at 25 µg/L using the primary source standards on a daily basis before sample analysis. Also run a CCV every 12 hours during an analytical sequence. Acceptance criteria are listed in Section 11.4.

10.6 A method blank must be prepared with each batch of samples not to exceed 12 hours. The method blank should be prepared from organic-free water. The method blanks are to be related to each 12-hour sequence of samples injected. Acceptance criteria for these blanks are listed in Section 8.2.4 and corrective action is listed in Section 11.5. Instrument blanks may be injected at any time in the sequence to verify absence of contamination.

10.7 A Blank spike/Blank spike duplicate (BS/BSD) must be prepared and analyzed with each batch of samples. The BS/BSDs are prepared using a source different than that used to prepare the initial calibration standards and contain all method target analytes (see 7.2.3.1 and 7.2.7). Acceptance criterion is 70-130% and ≤25% RPD. ***Refer to Attachment E for Mass DEP MCP requirements. See Table 3 for DoD blank spike acceptance criteria. Sporadic Marginal Exceedance number is as follows:***

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Number of Analytes in BS	Allowable number of Marginal Exceedances
>90	5
71-90	4
51-70	3
31-50	2
11-30	1
<11	0

Exceedances should be within marginal exceedance limits (Table 3) or expanded criterion listed above.

NOTE: The CCV and BS are considered equivalent for aqueous samples. Only one will need to be analyzed but it must be prepared from a source different than that used to perform initial calibration. In the case of solids, the BS is extracted similar to the samples. Use CCV acceptance criteria as presented in Section 8.2.2.

10.7.1 Control charts will be maintained for the BS/BSD for a representative subset of target analytes and surrogate spikes, in accordance with SOP 110.0014.

10.8 On an ongoing basis the laboratory analyzes matrix spikes and matrix spike duplicates from each batch of 20 samples.

10.8.1 The matrix spike is prepared as described in Section 7.2.9.

10.8.2 Matrix spike control limits are 70-130%. %RPD between spike and spike duplicate should be ≤30% (≤ 20% for AFCEE). ***For DoD matrix spike acceptance criteria use the blank spike acceptance criteria in Table 3. Refer to Attachment E for Mass DEP MCP requirements*** For samples outside control limits, see Section 11.6 for corrective action.

10.9 Surrogates recoveries limits are 70-130%. ***For DoD surrogate acceptance criteria use the blank spike acceptance criteria in Table 3. Refer to Attachment E for Mass DEP MCP requirements.*** For samples outside control limits, see Section 11.7 for corrective action.

10.10 The relative retention times (RRTs) need to be checked for each identified compound in samples, and compared to standard RRT. Acceptance criteria are presented in Section 11.8.

10.11 Ion abundance for target compounds and any tentatively identified compounds need to meet specific requirements. Acceptance criteria for ion abundance are presented in Section 11.9.

10.12 Internal standard area counts for standards and samples must meet specifications as described in Section 11.10.

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10.13 Data shall be checked to ascertain if it conforms to accepted practices. All sample analytical results used for final data reporting must be between the low standard and the high standard. Results, which fall below the low standard or above the high standard, are to be reported as estimated values. Corrective actions are described in Section 11.11.

10.14 MDLs are determined in reagent water or organic-free sand/soil and verified annually per SOP 110.0013. (Project-specific requirements may require that the MDL study be performed in the site-specific matrix.).

11.0 DATA VALIDATION

The items shall be verified and documented on the VOA Department Analysis Map (Attachment D.)

11.1 Ensure that the BFB tune was run at the beginning of each 12-hour sequence for each batch of samples analyzed. The acceptance criteria are listed below:

BFB Key Ions and Ion Abundance Criteria:

Ion	Abundance
50	15.0-40.0% of mass 95
75	30.0-60.0% of mass 95
95	base peak, 100% relative abundance
96	5.0-9.0% of mass 95
173	< 2.0% if mass 174
174	> 50.0% of mass 95
175	5.0-9.0% of mass 174
176	> 95.0%, but less than 101% of mass 174
177	5.0-9.0% of mass 176

11.1.1 If BFB acceptance criteria are not met, perform any or all of the following corrective actions:

Re-inject BFB.
Retune with PFTBA, then re-inject BFB.
Clean MS source, retune with PFTBA, and re-inject BFB.

11.1.2 If the tuning criteria still cannot be met after performing the above, have the mass spectrometer serviced by manufacturer representative.

11.2 After a seven-point initial calibration curve is analyzed, ensure that the following criteria were met.

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11.2.1 For the RFs, the %RSD must be less than 30% for all method target analytes, except for the poor purgers where the %RSD must be less than 50%.

11.2.1.1 If the %RSD of any method target analyte is 15% or less, then the relative response factor is assumed to be constant over the calibration range, and the average relative response factor may be used for quantitation.

11.2.1.2 If the %RSD of any method target analyte is greater than 15%, calibration curves must be constructed using first or higher order regression fits of the six calibration points. ***NOTE: A minimum of 5 points are used for average and linear regression calibration, six point for second order, and seven points for third order calibrations.*** The corresponding Coefficients of Determination (R^2) must be 0.99 or greater.

11.2.1.3 If these acceptance criteria are not met, then the following corrective actions should be performed: (1) adjust the instrument and/or perform instrument maintenance; or (2) narrow the calibration range using seven standards at different concentrations. The low end of the calibration curves must be carefully watched.

11.2.2 System Performance Check Compounds (SPCCs) and continuing calibration compounds (CCCs) must be evaluated and passed for initial calibration. Refer to Section 8.1.9 and 8.1.10.

11.3 If an initial calibration was performed, verify that initial calibration verification (ICV) was also performed. The percent difference for the CCC analytes must be within 20% (30% for all other target analytes). ***When samples are to be to be run for the DOD, all compounds must have ≤20% drift with no allowance.*** If not, reanalyze the ICV or prepare a new calibration curve as necessary.

11.4 Ensure the continuing calibration verification (CCV) standard is analyzed at the required frequency (every 12 hours or initially before daily analysis). In addition, the following acceptance criteria must be met.

11.4.1 System Performance Check Compounds (SPCCs): A system performance check must be made during every 12-hour shift. This is the same check that is applied during the initial calibration. If the minimum response factors are not met, the system must be evaluated, and corrective action must be taken before sample analysis begins. The minimum RF for volatile SPCCs is listed in Section 8.1.9.

11.4.2 Calibration Check Compounds (CCCs): After the system performance check is met, CCCs listed in Section 8.1.9 are used to check the validity of the initial calibration with a limit of <20% drift or difference. Acceptance criteria is ≤ 30% drift for all other method target analytes (50% for the poor

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purgers). *When samples are to be to be run for DOD, all compounds must have <20% drift).*

If these criteria are exceeded, corrective action is necessary. If corrective action fails to produce consecutive (immediate) calibration verification within acceptance criteria, then the analyst may perform maintenance to try to troubleshoot the instrument. After maintenance, the analyst must analyze two consecutive CCV at two concentration (one at or below mid-range) to demonstrate that corrective action was successful. If not, then a new six-point calibration must be generated. These criteria must be met before sample analysis begins and re-analysis of samples up to the last acceptable CCV standard must occur with the following exceptions:

- When the acceptance criteria is exceeded high, high bias, then all samples that are non-detects may be reported.

- When the acceptance criteria for the CCV are exceeded low, low bias, sample results that exceed a maximum regulatory limit may be reported.

11.4.3 When performing analyses for MCP or DOD, any analyte outside of criteria in the CCV must be noted in the project narrative

11.5 Assess the method blanks. The analyst shall confirm that this blank was analyzed at the required frequency and the criteria in Section 8.3.4 are met. Analytical batches with Method blanks outside acceptance criteria will be re-prepped and re-analyzed with the following exceptions:

11.5.1 Samples that are that are at least twenty times higher than the method blank may be reported.

11.5.2 When the method blank is less than 5% of the regulatory limit associated with the analyte the method blank would be acceptable.

11.5.3 If the analyte is found in the method blank above ½ the MRL but is not in any of the associated samples, no corrective action is needed.

11.5.4 Any results that are reported with method blank contamination must be B-flagged.

11.6 Assess that matrix spike/matrix spike duplicates were analyzed at required frequency. Acceptance criteria are that all % Recovery and/or RPD results meet project-established goals. If no project goals are specified, then results must be

within the control limits listed in Section 10. If results are not within criteria, perform the following corrective actions as appropriate.

11.6.1 If both BS/BSD and MS/MSD recoveries are unacceptable, then the entire batch of field and QC samples must be reanalyzed.

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11.6.2 If the MS/MSD is unacceptable, but the BS is acceptable, then a potential matrix effect has been identified. Reasonable attempts must be made to address matrix interference. The client must be notified in the case narrative of the matrix problem.

11.7 Check the surrogate calculations for correctness for all samples, blanks, BS, MS, and MSD. The following acceptance criteria apply to surrogate recoveries.

11.7.1 The surrogate recoveries for all QC samples must be within control limits of 70-130%. If the BS/BSD and/or method blank recoveries are outside limits, re-analysis must be performed for verification. If still outside limits, then corrective action is necessary. ***See Table 3 for surrogate acceptance limits for DoD projects.***

11.7.2 If sample surrogate recoveries exceed these limits, the sample must be re-analyzed. If the results are still outside the limits, then matrix affect has been verified and results are discussed in case narrative. ***NOTE: If an obvious matrix effect is seen on the chromatogram (Unresolved complex mixture, UCM), then re-analysis is not necessary. MA-MCP and CT-RCP projects must have a copy of the chromatogram included with the report.***

11.8 The relative retention times must be checked for all identified compounds in both standards and samples. The internal standard absolute retention times must also be checked for all analyses. Acceptance criteria are as follows:

11.8.1 The relative retention times of each compound in each calibration run should agree within 0.06 relative retention time (RRT) units. Late-eluting compounds usually have much better agreement.

11.8.2 Internal standard retention time:

11.8.2.1 If the retention time for any CCV internal standard changes by more than 30 seconds from the **mid-point standard of the initial calibration**, the chromatographic system must be inspected for malfunctions and corrections must be made, as required.

11.8.3 If the retention time for any sample internal standard changes by more than 30 seconds from the daily calibration, the chromatographic system must be inspected for malfunctions and corrections must be made, as required.

11.9 The analyst must verify that ion abundance meets specific criterion for the various analyses. The following acceptance criteria shall be checked for all appropriate samples.

11.9.1 All ions present in the standard mass spectrum at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) must be present in the sample spectrum.

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11.9.2 The relative intensities of ions specified in Section 11.10.1 must agree within plus or minus 30% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample abundance must be between 20 and 80 percent.)

11.9.2.1 Molecular ions present in the reference spectrum should be present in the sample spectrum.

11.9.2.2 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.

11.9.2.3 Ions present in the reference spectrum, but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

11.9.3 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. When serving the role as QA (or referee) laboratory, tentatively identified compounds (TICs) are always reported. Computer-generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. For example, the RCRA permit or waste de-listing requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches will the mass spectral interpretation specialist assign a tentative identification. Guidelines for making tentative identification are as follows:

11.9.3.1 Relative intensities of major ions in the reference spectrum (ions > 10% of the most abundant ion) should be present in the sample spectrum.

11.9.3.2 The relative intensities of the major ions should agree within ±30%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 20 and 80%.)

11.9.3.3 Molecular ions present in the reference spectrum should be present in the sample spectrum. Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.

11.9.3.4 Ions present in the reference spectrum, but not in the sample

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spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

11.10 The analyst will check the internal standard area counts for all calibration standards, QC samples, and samples for quantitation. If the area for any of the CCV internal standards changes by a factor of two (-50% to +100%) from the mid-point standard of the initial calibration, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. All sample internal standards must be -50 to 200% if their associated CCV. If the internal standard area counts fail this criterion, the following corrective actions should be considered:

11.10.1 Check to ensure there was no error in internal standards preparation or addition. Also, check instrument performance.

11.10.2 If the CCV failed criterion, re-analyze once. If this second consecutive CCV still does not meet criterion, then a new initial calibration must be performed.

11.10.3 If the internal standards for samples are outside criterion, then re-analyze once. If re-analysis produces results within criteria, then report these results. If the re-analysis is still outside criterion, discuss matrix issue in project narrative. ***NOTE: If an obvious matrix effect is displayed on the chromatogram (Unresolved complex mixture, UCM), then re-analysis is not necessary. MA-MCP and CT-RCP projects must have a copy of the chromatogram included with the report.***

11.11 The analyst must verify all reported results are derived from analytical results that are below the highest standard of the initial calibration curve and above the low standard. Values reported below the low standard are to be reported as estimated values (J values). For samples that exceed the calibration curve, dilute and analyze an appropriate sample aliquot.

11.12 All manual integration must be printed, when made, for verification.

11.13 Besides the items listed in Sections 11.1 through 11.12, the analyst should also verify the additional items as noted in Attachment D. A second level of review must be performed by a second analyst, results are recorded on Attachment D.

12.0 REFERENCES

12.1 SW-846 Method 8260B, Test Methods for the Analysis of Solid Waste, Third Edition, Update III.

12.2 HP GC EnviroQuant User's Guide, HPG1045A.

12.3 HP Environmental Data Analysis User's Guide HPG0032C.

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12.4 HP 6890 and 5890 Series II GC Operations Manuals.

12.5 HP 5971A and 5973 MS Operations Manuals.

12.6 NELAC Quality Systems Chapter 5, June 2003

12.7 DoD Quality Systems Manual, Final Version 4.2, October 2010.

13.0 ATTACHMENTS

13.1 Attachment A – Primary Standard Certificates of Analysis

13.2 Attachment B – Enviroquant method

13.3 Attachment C – Run logbook

13.4 Attachment D – Analysis Map

13.5 Attachment E – Massachusetts DEP WSC-CAM IIA requirements.

14.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

14.1 ESS Laboratory's policies on pollution prevention and waste management are covered in SOP 90_0002, Hazardous Waste Contingency and Emergency Response Plan. All employees are trained in the requirements of the SOP.

15.0 METHOD PERFORMANCE

15.1 Precision and Accuracy data must be generated by all employees before performing this analysis on client samples. The data is generated by analyzing a method blank and four blank spike samples. Acceptance criteria are 80-120% Recovery and %RSD of $\leq$ 20%. Five (5) sporadic marginal exceedances are allowed.

15.2 The precision and accuracy data in Table 1 were obtained using the SOP. Values are in ug/L.

16.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

Table 1. Typical Precision and Accuracy data generated 1/3/2005

Analyte	Spk	Avg	%Rec	%RSD	Analyte	Spk	Avg	%Rec	%RSD
Dichlorodifluoromethane	25	24.51	98	1.3	2-Hexanone	25	118.53	95	1.7
Chloromethane	25	23.82	95	1.1	trans-1,3-Dichloropropene	25	26.19	105	1.8
Vinyl Chloride	25	23.94	96	0.7	1,1,2-Trichloroethane	25	24.73	99	1.7
Bromomethane	25	25.37	101	1.9	1,3-Dichloropropane	25	25.19	101	0.9
Chloroethane	25	25.00	100	1.6	Tetrachloroethene	25	25.15	101	1.2

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Trichlorofluoromethane	25	25.05	100	0.2	Dibromochloromethane	25	27.05	108	1.3
Diethyl ether	25	24.63	99	1.7	1,2-Dibromoethane	25	25.26	101	1.4
Acetone	25	113.63	91	1.1	1-Chlorohexane	25	24.39	98	1.0
Carbon Disulfide	25	24.35	97	1.0	Chlorobenzene	25	24.75	99	1.3
1,1-Dichloroethene	25	24.37	97	0.5	1,1,1,2-Tetrachloroethane	25	25.23	101	1.1
Methylene Chloride	25	24.55	98	1.7	Ethylbenzene	25	24.67	99	1.0
Methyl tert-Butyl Ether	25	47.42	95	1.3	Xylene P,M	25	49.08	98	1.5
trans-1,2-Dichloroethene	25	24.42	98	2.4	Xylene O	25	24.56	98	1.8
1,1-Dichloroethane	25	24.31	97	1.8	Styrene	25	25.42	102	1.2
Vinyl Acetate	25	24.78	99	1.8	Bromoform	25	26.13	105	1.1
Di-isopropyl ether	25	24.71	99	1.8	Isopropylbenzene	25	24.86	99	0.8
Ethyl tertiary-butyl ether	25	24.92	100	1.6	1,2,3-Trichloropropane	25	24.13	97	1.2
2-Butanone	25	123.99	99	2.2	Bromobenzene	25	24.95	100	1.7
cis-1,2 Dichloroethene	25	24.54	98	1.6	1,1,2,2-Tetrachloroethane	25	24.79	99	1.9
2,2-Dichloropropane	25	24.47	98	2.1	n-Propylbenzene	25	24.56	98	1.9
Bromochloromethane	25	24.14	97	1.1	2-Chlorotoluene	25	24.31	97	3.9
Tetrahydrofuran	25	23.44	94	2.1	4-Chlorotoluene	25	24.20	97	1.1
Chloroform	25	24.53	98	1.4	1,3,5-Trimethylbenzene	25	24.53	98	1.6
1,1,1-Trichloroethane	25	24.92	100	1.1	tert-Butylbenzene	25	24.52	98	1.2
1,1-Dichloropropene	25	24.41	98	0.9	1,2,4-Trimethylbenzene	25	24.55	98	0.9
Carbon Tetrachloride	25	24.99	100	1.4	sec-Butylbenzene	25	24.67	99	1.3
Benzene	25	24.23	97	1.2	1,3 Dichlorobenzene	25	24.27	97	1.4
1,2-Dichloroethane	25	25.00	100	1.6	4-Isopropyltoluene	25	24.42	98	1.4
Tertiary-amyl methyl ether	25	24.64	99	1.8	1,4 Dichlorobenzene	25	24.46	98	1.4
Trichloroethene	25	24.46	98	1.3	n-Butylbenzene	25	24.65	99	1.0
1,2-Dichloropropane	25	24.25	97	1.8	1,2 Dichlorobenzene	25	24.23	97	1.1
Dibromomethane	25	24.61	98	2.1	1,2-Dibromo-3-Chloropropane	25	25.46	102	1.8
1,4-Dioxane	25	489.19	98	11.1	1,2,4-Trichlorobenzene	25	24.36	97	1.9
Bromodichloromethane	25	25.14	101	1.9	Hexachlorobutadiene	25	26.30	105	1.9
4-Methyl-2-Pentanone	25	121.18	97	1.7	Naphthalene	25	23.59	94	1.3
cis-1,3-Dichloropropene	25	25.30	101	1.7	1,2,3-Trichlorobenzene	25	23.71	95	2.3
Toluene	25	24.70	99	1.1					

17.0 DEFINITIONS

17.1 **Accuracy**: The closeness of agreement between an observed value and an accepted reference value. When applied to a set of observed values, accuracy will be a combination of a random component and of a common systematic error (or bias) component.

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17.2 **Batch**: A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 20, then each group of 20 samples or less will all be handled as a separate batch.

17.3 **Bias**: The deviation due to matrix effects of the measured value (x_s - x_u) from a known spiked amount, where x_s is the spiked sample and x_u is the un-spiked sample. Bias can be assessed by comparing a measured value to an accepted reference value in a sample of known concentration or by determining the recovery of a known amount of contaminant spiked into a sample (matrix spike).

17.4 **Control Sample**: A QC sample introduced into a process to monitor the performance of the system.

17.5 **Equipment Blank**: A sample of analyte-free media which has been used to rinse the sampling equipment. It is collected after completion of decontamination and prior to sampling. This blank is useful in documenting adequate decontamination of sampling equipment.

17.6 **Method Reporting Limit**: The lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The MRL is generally 5 to 10 times the MDL. ESS Laboratory sets the MRL to the lowest non-zero standard in the calibration curve or higher.

17.7 **Field Duplicates**: Independent samples which are collected as close as possible to the same point in space and time. They are two separate samples taken from the same source, stored in separate containers, and analyzed independently. These duplicates are useful in documenting the precision of the sampling process.

17.8 **Blank Spike (BS)**: A known matrix spiked with compound(s) representative of the target analytes. This is used to document laboratory performance.

17.9 **Matrix**: The component or substrate (e.g., surface water, drinking water) which contains the analyte of interest.

17.10 **Matrix Duplicate**: An intralaboratory split sample which is used to document the precision of a method in a given sample matrix.

17.11 **Matrix Spike**: An aliquot of sample spiked with a known concentration of target analyte(s). The spiking occurs prior to sample preparation and analysis. A matrix spike is used to document the bias of a method in a given sample matrix.

17.12 **Matrix Spike Duplicates**: Intralaboratory split samples spiked with identical concentrations of target analyte(s). The spiking occurs prior to sample preparation and analysis. They are used to document the precision and bias of a method in a given sample matrix.

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17.13 **Method Blank**: An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank should be carried through the complete sample preparation and analytical procedure. The method blank is used to document contamination resulting from the analytical process.

17.14 **Method Detection Limit (MDL)**: The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix type containing the analyte. See SOP 110_0013 for further explanation.

17.15 **Organic-Free Reagent Water**: For volatiles, all references to water in the method refer to water in which an interferant is not observed at the method detection limit of the compounds of interest. A water purification system is usedto generate organic-free deionized water.

17.16 **Records**: Include all logbooks, papers, machine readable materials, or other documentary materials, regardless of physical form or characteristics.

17.17 **Surrogate**: An organic compound which is similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which is not normally found in environmental samples.

17.18 **Trip Blank**: A sample of analyte-free media taken from the laboratory to the sampling site and returned to the laboratory unopened. A trip blank is used to document contamination attributable to shipping and field handling procedures. This type of blank is useful in documenting contamination of volatile organics samples.

18.0 PERSONNEL QUALIFICATIONS

18.1 Analysts who perform this analysis must have a working knowledge or quantitative and qualitative analysis, instrumental methods of analysis, chemical laboratory methods, and equipment.

18.2 All analysts, before performing any analysis, participate in the ESS Laboratory training program (SOP80_0016). The training process consists of reading the Standard Operating Procedure, gaining instruction on the procedure from an experienced analyst, and performing the initial demonstration of capability.

19.0 TROUBLESHOOTING

19.1 If BFB criteria are not met, the analysis must be repeated. Analysis can not begin until BFB meets acceptance criteria. Repeated failures indicate that the MS acquisition parameters must be adjusted. These parameters should be adjusted in manual tune, saved and the tune repeated.

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19.2 If manual or auto-tune does not produce BFB spectra within acceptance criteria, the MS source may need cleaning. Cleaning instructions are found in the MS detector manual.

19.3 Method blanks must not contain any target compound greater than ½ the MRL with the exception of methylene chloride (see section 11.5 for expanded criteria). If the method blank does not meet criteria, then system must be cleaned before processing samples. This includes washing the purge vessel and baking the trap.

19.4 See laboratory supervisor or operations manager for all other maintenance problems.

19.5 Record all maintenance in the instrument's maintenance logbook.

20.0 Data Management And Records

20.1 **Data Management** - ESS Laboratory's utilizes the Promium Element LIMS system as part of its Data Management system. Client sample information is entered into ELEMENT LIMS and analyses are assigned to each sample. The LIMS allows EPA hold times, minimum batch QC requirements, and QC criteria to be assigned to each analysis. Standards can be entered and assigned to QC samples through the LIMS. Once analysis has been performed, data is imported using DataTool avoiding manual errors. In conjunction with Crystal Reports, the ELEMENT system allows for a wide variety of reporting formats.

20.2 **Records** – The specific retention periods required in the NELAC Standards, EPA-CFR and state and local statutes are followed or exceeded. At a minimum, data records are retained for five years from last use (10 years for drinking water). If there is a question about whether a record should be retained or disposed because no specific requirement could be found, the record is retained until such time as a retention period is specified. Records are stored in specified-labeled locations and are easily retrievable. All raw data associated with testing is also retained including; computer printouts, chromatograms, review forms, and logbooks.

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TABLE 2 -Internal Standards with their associated compounds

PENTAFLUOROBENZENE (IS)	CHLOROBENZENE-d5 (IS)	1,4-DICHLOROBENZENE-d4 (IS)
Dichlorodifluoromethane	Toluene - d_8	Isopropylbenzene
Chloromethane	Toluene	trans-1,4-Dichloro-2-Butene
Vinyl Chloride	2-Hexanone (P)	1,2,3-Trichloropropane
Bromomethane	Ethyl methacrylate	Bromobenzene
Chloroethane (P)	1,3-Dichloropropane	1,1,2,2-Tetrachloroethane
Trichlorofluoromethane	Tetrachloroethene	n-Propylbenzene
Diethyl Ether	Dibromochloromethane	2-Chlorotoluene
Acrolein (P)	1,2-Dibromoethane	4-Chlorotoluene
1,1,2-Trichloro-1,2,2-trifluoroethane	1-Chlorohexane	1,3,5-Trimethylbenzene
Acetone (P)	Chlorobenzene	tert-Butylbenzene
Iodomethane (P)	1,1,1,2-Tetrachloroethane	Pentachloroethane
Carbon Disulfide (P)	Ethylbenzene	1,2,4-Trimethylbenzene
1,1-Dichloroethene	Xylene, M,O	sec-Butylbenzene
Allyl Chloride	Xylene P	1,3-Dichlorobenzene
Methyl acetate	Styrene	4-Isopropyltoluene
Methylene Chloride	Bromoform	1,4-Dichlorobenzene
Methyl tert-butyl ether	cis 1,4-Dicchloro-2-butene	n-Butylbenzene
Acrylonitrile (P)	4-Bromofluorobenzene*	1,2-Dichlorobenzene
trans-1,2-Dichloroethene		Hexachloroethane
1,1-Dichloroethane		1,2-Dibromo-3-chloropropane (P)
Vinyl Acetate		1,2,4-Trichlorobenzene
Chloroprene		Hexachlorobutadiene
Di-isopropyl ether		Naphtalene
Ethyl tertiary butyl ether		1,2,3-Trichlorobenzene
2-Butanone (P)		
cis-1,2-Dichloroethene		
2,2-Dichloropropane		
Methyl acrylate		
Bromochloromethane		
Methacrylonitrile		
Tetrahydrofuran		
Chloroform		
Dibromofluoromethane*		
1,1,1-Trichloroethane		
Cyclohexane		
1-Chlorobutane		
1,1-Dichloropropene		
Carbon Tetrachloride		
1,2-Dichloroethane-D4*		
Benzene		
1,2-Dichloroethane		
Tertiary-amyl methyl ether		
Trichlorethene		
Methylcyclohexane		
1,2-Dichloropropane		
Dibromomethane		
1,4-Dioxane (P)		
Bromodichloromethane		
2-Nitropropane		
2-Chloroethyl vinyl ether (P)		
4-Methyl-2-pentanone (P)		
cis-1,3-Dichloropropene		
Toluene		
trans-1,3-Dichloropropene		
1,1,2-Trichloroethane		
IS = Internal Standard; * = Surrogate; (P) = Poor purger		

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Table 3 (DoD Quality Systems Manual Version 4.1, Table G-4) Blank Spike QC Limits

	DoD			
Analyte	LCL	UCL	LMEL	UMEL
1,1,1,2-Tetrachloroethane	80	130	75	135
1,1,1-Trichloroethane	65	130	55	145
1,1,2,2-Tetrachloroethane	65	130	55	140
1,1,2-Trichloroethane	75	125	65	135
1,1-Dichloroethane	70	135	60	145
1,1-Dichloroethene	70	130	55	140
1,1-Dichloropropene	75	130	65	140
1,2,3-Trichlorobenzene	55	140	45	155
1,2,3-Trichloropropane	75	125	65	130
1,2,4-Trichlorobenzene	65	135	55	145
1,2,4-Trimethylbenzene	75	130	65	140
1,2-Dibromo-3-Chloropropane	50	130	35	145
1,2-Dibromoethane	80	120	75	125
1,2-Dichlorobenzene	70	120	60	130
1,2-Dichloroethane	70	130	60	140
1,2-Dichloropropane	75	125	65	135
1,3,5-Trimethylbenzene	75	130	65	140
1,3 Dichlorobenzene	75	125	65	130
1,3-Dichloropropane	75	125	65	135
1,4 Dichlorobenzene	75	125	65	130
2,2-Dichloropropane	75	135	60	150
2-Butanone	30	150	10	170
2-Chlorotoluene	75	125	65	135
2-Hexanone	55	130	45	140
4-Chlorotoluene	75	130	65	135
4-Methyl-2-Pentanone	60	135	45	145
Acetone	40	140	20	160
Benzene	80	120	75	130
Bromobenzene	75	125	70	130
Bromochloromethane	65	130	55	140
Bromodichloromethane	75	120	70	130
Bromoform	70	130	60	140
Bromomethane	30	145	10	165
Carbon Disulfide	35	160	15	185
Carbon Tetrachloride	65	140	55	150
Chlorobenzene	80	120	75	130
Dibromochloromethane	60	135	45	145
Chloroethane	60	135	50	145
Chloroform	65	135	50	150

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Chloromethane	40	125	25	140
cis-1,2 Dichloroethene	70	125	60	135
cis-1,3-Dichloropropene	70	130	60	140
Dibromomethane	75	125	65	135
Dichlorodifluoromethane	30	155	10	175
Ethylbenzene	75	125	65	135
Hexachlorobutadiene	50	140	35	160
Isopropylbenzene	75	125	65	135
m,p-Xylene	75	130	65	135
Methylene Chloride	55	140	40	155
Methyl tert-Butyl ether	65	125	55	135
Naphthalene	55	140	40	150
n-Butylbenzene	70	135	55	150
n-Propylbenzene	70	130	65	140
o-Xylene	80	120	75	130
p-Isopropyltoluene	75	130	65	140
sec-Butylbenzene	70	125	65	135
Styrene	65	135	55	145
tert-Butylbenzene	70	130	60	140
Tetrachloroethene	45	150	25	165
Toluene	75	120	70	130
trans-1,2-Dichloroethene	60	140	45	150
trans-1,3-Dichloropropene	55	140	40	155
Trichloroethene	70	125	60	135
Trichlorofluoromethane	60	145	45	160
Vinyl Chloride	50	145	35	165
Surrogates:				
Dibromofluoromethane	85	115	-	-
Toluene-d8	85	120	-	-
4-Bromofluorobenzene	75	120	-	-
1,2-Dichloroehane-d4	70	120	-	-

LCL = Lower Control Limit
UCL = Upper Control Limit
LMEL = Lower Marginal Exceedance Limit
UMEL = Upper Marginal Exceedance Limit

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Table 4

Summary of Method Quality Objectives for Method 8260B Volatile Organic Compounds

QC Element	Frequency	Target Analyte/Surrogate	Corrective action
Tune	Every twelve hours	Criteria in Table 1	Suspend analysis until criteria met.
Initial Calibration Minimum 5-point ICAL for all analytes	Instrument set up. Each time the ICV or CCV can not meet criteria.	RSD≤15%, R≥0.995,R^2≥0.99 Do not force through origin	Recalibrate as required.
ICV	Immediately following initial calibration.	%Rec = 70-130% ***DOD : 80-120%***	Re-analyze once, if still fails criteria re-calibrate instrument.
CCV	At the beginning of each analytical sequence. No more than 20 samples or 12 hours between CCVs.	CCCs must be < 20%, all other analytes < 30% drift or difference. ***DoD : 80-120% all compounds.***	Re-analyze once, if still fails criteria re-calibrate instrument.
Blank spike/ Blank spike duplicate	At the beginning of each analytical sequence. Prepared using source different than that used to prepare initial calibration standards and contain all target analytes.	All target analytes < 30% drift or difference and ≤ 25%RPD, except for 1,4-dioxane (screen), where lab CLs are used. ***allows for 5 sporadic marginal failures. See Table 3 for DoD criteria***	Re-analyze once with associated samples. If still outside criteria, report exceedance in project narrative. The lab is to identify those difficult analyte that routinely are outside the 70-130% criterion, disclose in P/N.
Method Blank	One per analytical batch of 20 or fewer samples. Use second source standard	Analytes < ½ MRL	See Section 11.0. Report nonconformance in case narrative.
MS/MSD	1 per 20 samples.	% Rec = 70-130%, except for 1,4-dioxane (screen), where lab CLs are used; ≤ 30% RPD . ***See Table 3 for DoD criteria***	Check BS, if BS is within criteria then report non-conformance in project narrative. If BS fails, then re-analyze.
Surrogates	Added to all samples and standards.	%Rec = 70-130% ***See Table 3 for DoD criteria***	If one or more is outside criteria, then re-analyze sample unless an obvious interference is present (UCM).
Internal Standards	Added to all samples and standards.	50-200% of area counts in the associated continuing calibration standard.	If one or more is outside criteria, then re-analyze sample unless an obvious interference is present (UCM).

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5B14014
5B14015
5B14016

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Tel: (800) 356-1688
Fax: (814) 353-1309

Catalog No.: 30006 **Lot No.:** A034309

Description: VOA Calibration Mix #1

Expiration Date[1]: February 2008 **Storage:** Freezer

Elution Order	Compound	CAS#	Percent Purity[2]	Concentration[3]	Percent Uncertainty[4]
1	Acetone	67-64-1	99%	5000 ug/mL	+/- 0.1
2	2-Butanone (MEK)	78-93-3	99%	5000 ug/mL	+/- 0.1
3	4-Methyl-2-pentanone (MIBK)	108-10-1	99%	5000 ug/mL	+/- 0.1
4	2-Hexanone	591-78-6	99%	5000 ug/mL	+/- 0.1

Solvent: P&T Methanol/Water (90:10) 67-56-1/7732-18-5 99%

Column:
105m x .32mm x 3.0um
Rtx-502.2 (cat.#10910)

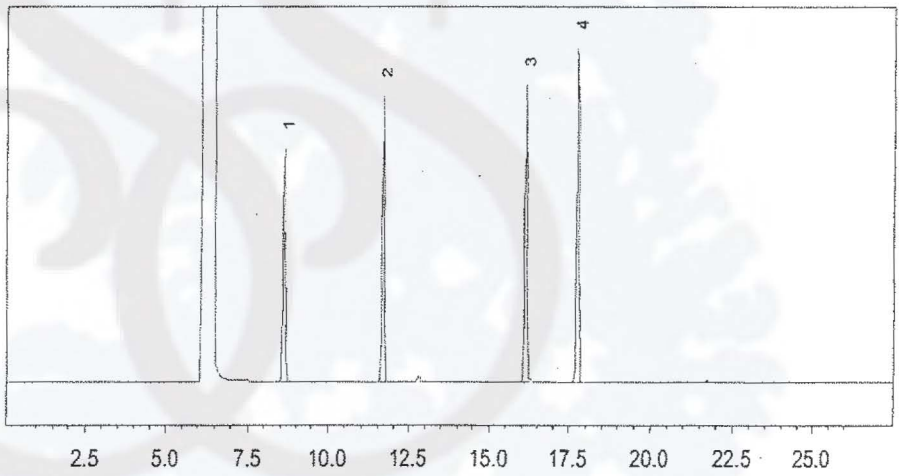
Carrier Gas:
hydrogen @ 40 cc/min.

Temp. Program:
40°C (hold 2 min.) to 240°C @ 8°C/min.

Inj. Temp:
200°C

Det. Temp:
250°C

Det. Type:
FID



Manufactured By: MEW

John Lidgett - QA Analyst

1 Expiration date of the unopened ampul stored at recommended temperature.
2 Purity was determined by one or more of the following techniques: GC/FID, HPLC, GC/ECD, GC/MS. Value rounded to the nearest LOWER whole percentage. In addtion to detectors listed above, chemical identity and purity are confirmed using 1 or more of the following: MS, DSC, solid probe MS, GC/FPD, GC/NPD, GC/TC, FTIR, melting point, reftactive index, and Karl Fisher. See data pack or contact Restek for further details.
3 Based upon gravimetric preperation with balance calibration verified using NISTtraceable weights (7 mass levels).
4 Percent Uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.

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Tel: (800) 356-1688
Fax: (814) 353-1309

Catalog No.: 30042 **Lot No.:** A034239

Description: 502.2 Calibration Mix #1

Expiration Date[1]: July 2011 **Storage:** Freezer

Elution Order	Compound	CAS#	Percent Purity[2]	Concentration[3]	Percent Uncertainty[4]
1	Dichlorodifluoromethane (CFC-12)	75-71-8	99%	2003 ug/mL	+/- 1.3
2	Chloromethane (methyl chloride)	74-87-3	99%	1997 ug/mL	+/- 1.3
3	Vinyl chloride	75-01-4	99%	2002 ug/mL	+/- 2.1
4	Bromomethane (methyl bromide)	74-83-9	99%	1995 ug/mL	+/- 1.3
5	Chloroethane (ethyl chloride)	75-00-3	99%	1996 ug/mL	+/- 1.3
6	Trichlorofluoromethane (CFC-11)	75-69-4	99%	2000 ug/mL	+/- 0.3
	Solvent: P&T Methanol	67-56-1	99%		

Column:
105m x .32mm x 1.8um
Rtx-502.2 (cat.#10921)

Carrier Gas:
helium @ 2.2 ml/min.

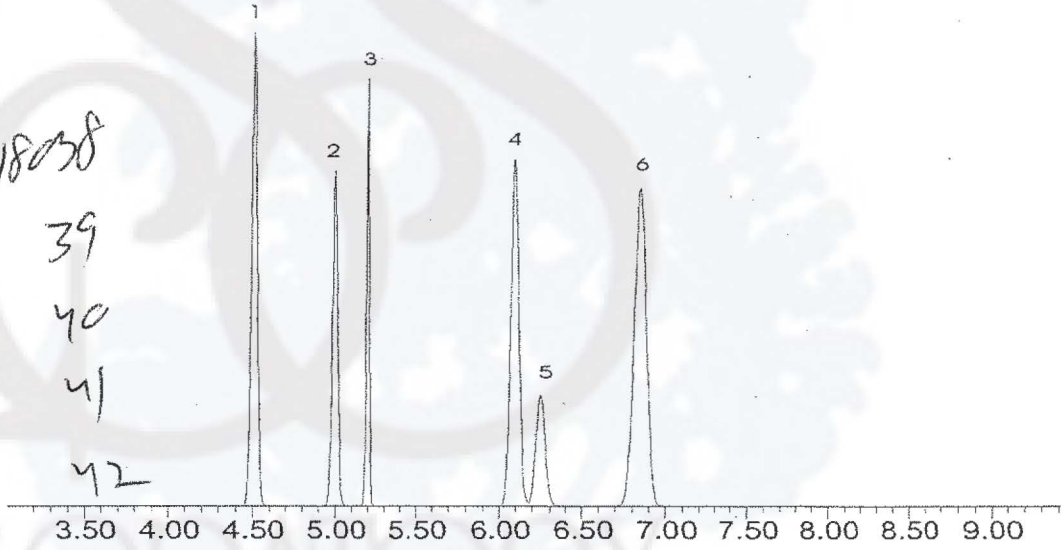
Temp. Program:
50°C (hold 9.5 min.) to 50°C @ 8°C/min.

Inj. Temp:
200°C

Det. Temp:
250°C

Det. Type:
MSD

5C18038
39
40
41
42



Manufactured By: MEW

John Lidgett - QA Analyst

1 Expiration date of the unopened ampul stored at recommended temperature.
2 Purity was determined by one or more of the following techniques: GC/FID, HPLC, GC/ECD, GC/MS. Value rounded to the nearest LOWER whole percentage. In addtion to detectors listed above, chemical identity and purity are confirmed using 1 or more of the following: MS, DSC, solid probe MS, GC/FPD, GC/NPD, GC/TC, FTIR, melting point, reftactive index, and Karl Fisher. See data pack or contact Restek for further details.
3 Based upon gravimetric preperation with balance calibration verified using NISTtraceable weights (7 mass levels).
4 Percent Uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.

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Bellefonte, PA 16823-8812
Tel: (800) 356-1688
Fax: (814) 353-1309

Catalog No.: 558360 **Lot No.:** A036277

Description: Custom VOA Additions Standard

Expiration Date[1]: October 2006 **Storage:** Freezer

Elution Order	Compound	CAS#	Percent Purity[2]	Concentration[3]	Percent Uncertainty[4]
1	Methyl acetate	79-20-9	99%	2000 ug/mL	+/- 0.1
2	1-Chlorobutane (Butyl chloride)	109-69-3	99%	2000 ug/mL	+/- 0.1
3	Cyclohexane	110-82-7	99%	2000 ug/mL	+/- 0.1
4	Methylcyclohexane	108-87-2	99%	2000 ug/mL	+/- 0.1
5	1-Chlorohexane	544-10-5	99%	2000 ug/mL	+/- 0.1
6	Hexachloroethane	67-72-1	99%	2000 ug/mL	+/- 0.1
	Solvent: P&T Methanol	67-56-1	99%		

Column:
105m x .32mm x 1.8um
Rtx-502.2 (cat.#10921)

Carrier Gas:
helium @ 2.2 ml/min.

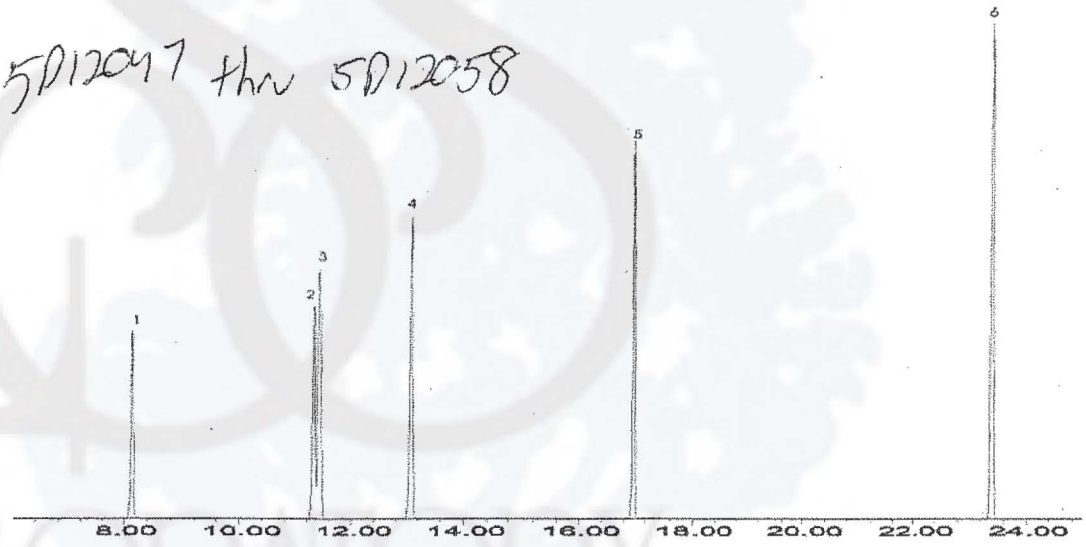
Temp. Program:
40°C (hold 2 min.) to 240°C
@ 8°C/min. (hold 10 min.)

Inj. Temp:
200°C

Det. Temp:
250°C

Det. Type:
MSD

5D12047 thru 5D12058



Manufactured By: JP

John Lidgett

John Lidgett - QA Analyst

1 Expiration date of the unopened ampul stored at recommended temperature.
2 Purity was determined by one or more of the following techniques: GC/FID, HPLC, GC/ECD, GC/MS. Value rounded to the nearest LOWER whole percentage. In addtion to detectors listed above, chemical identity and purity are confirmed using 1 or more of the following: MS, DSC, solid probe MS, GC/FPD, GC/NPD, GC/TC, FTIR, melting point, reftactive index, and Karl Fisher. See data pack or contact Restek for further details.
3 Based upon gravimetric preperation with balance calibration verified using NISTtraceable weights (7 mass levels).
4 Percent Uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.

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Bellefonte, PA 16823-8812
Tel: (800) 356-1688
Fax: (814) 353-1309

Catalog No.: 30475A Lot No.: A036598

Description: 8260B Calibration Mix #1

Expiration Date[1]: April 2008 Storage: Freezer

5E 2068 by 7076

Elution Order	Compound	CAS#	Percent Purity[2]	Concentration[3]	Percent Uncertainty[4]
1	Diethyl ether (ethyl ether)	60-29-7	99%	2000 ug/mL	+/- 0.3
2	1,1,2-Trichlorotrifluoroethane (CFC-113)	76-13-1	99%	2000 ug/mL	+/- 0.3
3	1,1-Dichloroethylene	75-35-4	99%	2000 ug/mL	+/- 0.3
4	Acetonitrile	75-05-8	99%	2000 ug/mL	+/- 0.3
5	Iodomethane (methyl iodide)	74-88-4	99%	2000 ug/mL	+/- 0.3
6	Allyl chloride (3-chloropropene)	107-05-1	99%	2000 ug/mL	+/- 0.3
7	Methylene chloride (dichloromethane)	75-09-2	99%	2000 ug/mL	+/- 0.3
8	Carbon disulfide	75-15-0	99%	2000 ug/mL	+/- 0.3
9	Acrylonitrile	107-13-1	99%	2000 ug/mL	+/- 0.3
10	trans-1,2-Dichloroethylene	156-60-5	99%	2000 ug/mL	+/- 0.3
11	1,1-Dichloroethane	75-34-3	98%	2000 ug/mL	+/- 0.3
12	Chloroprene (2-chloro-1,3-butadiene)	126-99-8	99%	1961 ug/mL	+/- 1.6
13	Propionitrile	107-12-0	99%	2000 ug/mL	+/- 0.3
14	2,2-Dichloropropane	594-20-7	99%	2000 ug/mL	+/- 0.3
15	cis-1,2-Dichloroethylene	156-59-2	98%	2000 ug/mL	+/- 0.3
16	Methacrylonitrile	126-98-7	99%	2000 ug/mL	+/- 0.3
17	Methyl acrylate	96-33-3	99%	2000 ug/mL	+/- 0.3
18	Isobutanol (2-Methyl-1-propanol)	78-83-1	99%	2000 ug/mL	+/- 0.3
19	Chloroform	67-66-3	99%	2000 ug/mL	+/- 0.3
20	Bromochloromethane	74-97-5	99%	2000 ug/mL	+/- 0.3
21	Tetrahydrofuran	109-99-9	99%	2000 ug/mL	+/- 0.3
22	1,1,1-Trichloroethane	71-55-6	99%	2000 ug/mL	+/- 0.3
23	1,1-Dichloropropene	563-58-6	99%	2000 ug/mL	+/- 0.3
24	Carbon tetrachloride	56-23-5	99%	2000 ug/mL	+/- 0.3
25	1,2-Dichloroethane	107-06-2	99%	2000 ug/mL	+/- 0.3
26	Benzene	71-43-2	99%	2000 ug/mL	+/- 0.3
27	2-Chloroethanol (ethylene chlorhydrin)	107-07-3	99%	2000 ug/mL	+/- 0.3
28	Trichloroethylene	79-01-6	99%	2000 ug/mL	+/- 0.3
29	1,2-Dichloropropane	78-87-5	98%	2000 ug/mL	+/- 0.3
30	Methyl methacrylate	80-62-6	99%	2000 ug/mL	+/- 0.3
31	Bromodichloromethane	75-27-4	99%	2000 ug/mL	+/- 0.3
32	1,4-Dioxane	123-91-1	99%	2000 ug/mL	+/- 0.3
33	2-Nitropropane	79-46-9	98%	2000 ug/mL	+/- 0.3
34	Dibromomethane	74-95-3	99%	2000 ug/mL	+/- 0.3
35	cis-1,3-Dichloropropylene	10061-01-5	99%	2000 ug/mL	+/- 0.3
36	Toluene	108-88-3	99%	2000 ug/mL	+/- 0.3
37	Ethyl methacrylate	97-63-2	99%	2000 ug/mL	+/- 0.3
38	trans-1,3-Dichloropropylene	10061-02-6	99%	2000 ug/mL	+/- 0.3
39	1,1,2-Trichloroethane	79-00-5	99%	2000 ug/mL	+/- 0.3
40	1,3-Dichloropropane	142-28-9	99%	2000 ug/mL	+/- 0.3
41	Tetrachloroethylene	127-18-4	99%	2000 ug/mL	+/- 0.3
42	Dibromochloromethane	124-48-1	99%	2000 ug/mL	+/- 0.3
43	1,2-Dibromoethane (EDB)	106-93-4	99%	2000 ug/mL	+/- 0.3
44	Chlorobenzene	108-90-7	99%	2000 ug/mL	+/- 0.3
45	1,1,1,2-Tetrachloroethane	630-20-6	99%	2000 ug/mL	+/- 0.3
46	Ethylbenzene	100-41-4	99%	2000 ug/mL	+/- 0.3
47	m-Xylene	108-38-3	99%	2000 ug/mL	+/- 0.3
48	p-Xylene	106-42-3	99%	2000 ug/mL	+/- 0.3
49	o-Xylene	95-47-6	99%	2000 ug/mL	+/- 0.3
50	Styrene	100-42-5	99%	2000 ug/mL	+/- 0.3
51	Isopropylbenzene (cumene)	98-82-8	99%	2000 ug/mL	+/- 0.3
52	Bromoform	75-25-2	99%	2000 ug/mL	+/- 0.3
53	cis-1,4-Dichloro-2-butene	1476-11-5	97%	2000 ug/mL	+/- 0.3
54	1,1,2,2-Tetrachloroethane	79-34-5	99%	2000 ug/mL	+/- 0.3
55	1,2,3-Trichloropropane	96-18-4	99%	2000 ug/mL	+/- 0.3
56	trans-1,4-dichloro-2-butene	110-57-6	98%	2000 ug/mL	+/- 0.3
57	n-Propylbenzene	103-65-1	99%	2000 ug/mL	+/- 0.3
58	Bromobenzene	108-86-1	99%	2000 ug/mL	+/- 0.3

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Bellefonte, PA 16823-8812
Tel: (800) 356-1688
Fax: (814) 353-1309

Catalog No.: 30216 **Lot No.:** A035108

Description: Vinyl Acetate Standard

Expiration Date[1]: December 2009 **Storage:** Freezer

Elution Order	Compound	CAS#	Percent Purity[2]	Concentration[3]	Percent Uncertainty[4]
1	Vinyl acetate	108-05-4	99%	2000 ug/mL	+/- 0.1
	Solvent: P&T Methanol	67-56-1	99%		

Column:
105m x .53mm x 3.0um
Rtx-502.2 (cat.#10910)

Carrier Gas:
hydrogen @ 40 cm/sec.

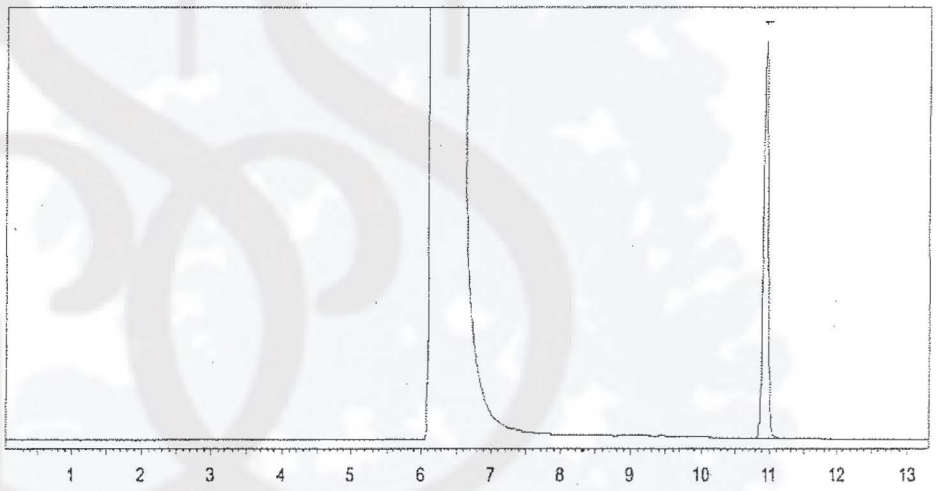
Temp. Program:
40°C (hold 2 min.) to 130°C @ 8°C/min.

Inj. Temp:
200°C

Det. Temp:
250°C

Det. Type:
FID

5E0071
72
73
74



Manufactured By: MEW

John Lidgett - QA Analyst

1 Expiration date of the unopened ampul stored at recommended temperature.
2 Purity was determined by one or more of the following techniques: GC/FID, HPLC, GC/ECD, GC/MS. Value rounded to the nearest LOWER whole percentage. In addtion to detectors listed above, chemical identity and purity are confirmed using 1 or more of the following: MS, DSC, solid probe MS, GC/FPD, GC/NPD, GC/TC, FTIR, melting point, reftactive index, and Karl Fisher. See data pack or contact Restek for further details.
3 Based upon gravimetric preperation with balance calibration verified using NISTtraceable weights (7 mass levels).
4 Percent Uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.

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Certificate #FM80397

Tech Tip:

Vinyl acetate is a volatile organic ester included in the target lists of several US EPA and other methods. Under acidic conditions, esters react with alcohols to form new esters (transesterification). Methanol-based mixes containing halogenated compounds are slightly acidic, so it is important to minimize exposure of vinyl acetate to mixes of halogenated compounds in methanol. For this reason, we offer vinyl acetate in individual solution, and suggest that it be introduced into the working level calibration solution immediately before use. This will minimize problems and ensure more consistent results.

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110 Benner Circle
Bellefonte, PA 16823-8812
Tel: (800) 356-1688
Fax: (814) 353-1309

Catalog No.: 30475B **Lot No.:** A031415

Description: 8260B Calibration Mix #2

Expiration Date[1]: January 2008 **Storage:** Freezer

Elution Order	Compound	CAS#	Percent Purity[2]	Concentration (weight/volume)[3]	Percent Uncertainty[4]
1	2-Chloroethyl vinyl ether	110-75-8	99%	2000 ug/mL	+/- 0.04
	Solvent: P&T Methanol	67-56-1	99%		

Column:
105m x .53mm x 3.0um
Rtx-502.2 (cat.#10910)

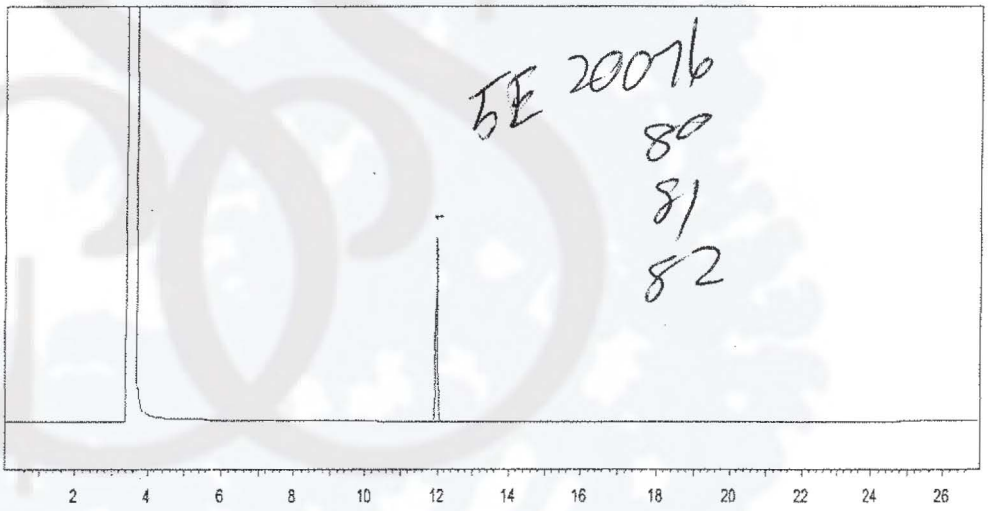
Carrier Gas:
hydrogen @ 40 cm/sec

Temp. Program:
40°C (hold 2 min.) to 240°C
@ 8°C/min.

Inj. Temp:
200°C

Det. Temp:
250°C

Det. Type:
FID



Manufactured By: JAM

John Lidgett - QA Analyst

1 Expiration date of the unopened ampul stored at recommended temperature.
2 Purity was determined by one or more of the following techniques: GC/FID, HPLC, GC/ECD, GC/MS. Value rounded to the nearest LOWER whole percentage. In addtion to detectors listed above, chemical identity and purity are confirmed using 1 or more of the following: MS, DSC, solid probe MS, GC/FPD, GC/NPD, GC/TC, FTIR, melting point, reftactive index, and Karl Fisher. See data pack or contact Restek for further details.
3 Based upon gravimetric preperation with balance calibration verified using NISTtraceable weights (7 mass levels).
4 Percent Uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.

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110 Benner Circle
Bellefonte, PA 16823-8812
Tel: (800) 356-1688
Fax: (814) 353-1309

Catalog No.: 30465 **Lot No.:** A031618

Description: California Oxygenates Mixture #1

Expiration Date[1]: April 2007 **Storage:** Freezer

Elution Order	Compound	CAS#	Percent Purity[2]	Concentration (weight/volume)[3]	Percent Uncertainty[4]
1	tert-Butanol (TBA)	75-65-0	99%	10000 ug/mL	+/- 0.1
2	Methyl-tert-butyl ether (MTBE)	1634-04-4	99%	2000 ug/mL	+/- 0.1
3	Diisopropyl ether (DIPE)	108-20-3	99%	2000 ug/mL	+/- 0.1
4	Ethyl-tert-butyl ether (ETBE)	637-92-3	99%	2000 ug/mL	+/- 0.1
5	tert-Amyl methyl ether (TAME)	994-05-8	99%	2000 ug/mL	+/- 0.1
	Solvent: P&T Methanol	67-56-1	99%		

Column:
105m x .32mm x 1.8um
Rtx-502.2 (cat.#10921)

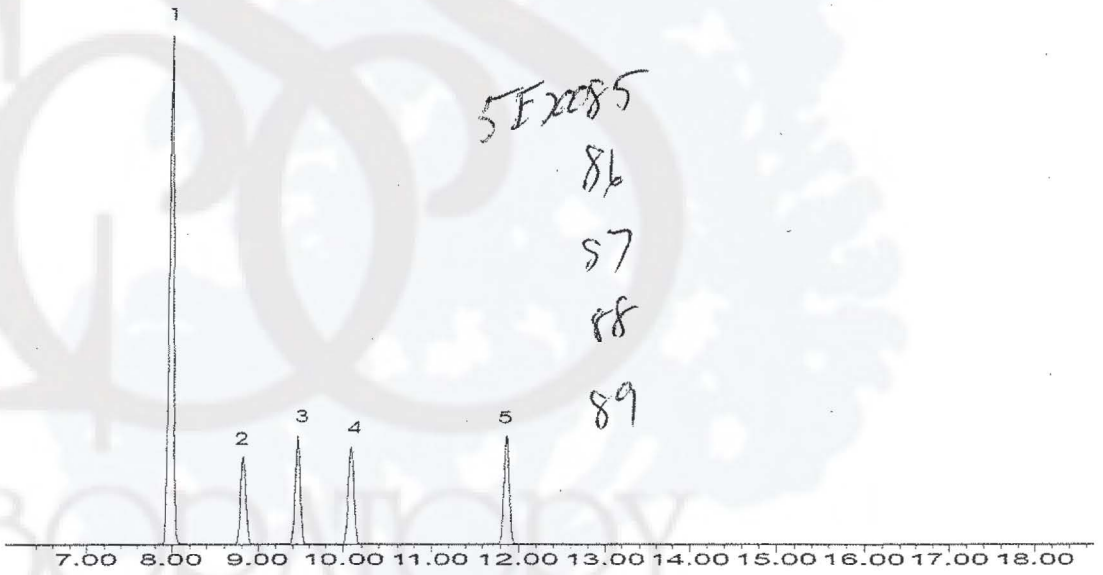
Carrier Gas:
helium @ 2.2 ml/min.

Temp. Program:
40°C (hold 2 min.) to 175°C
@ 8°C/min.

Inj. Temp:
200°C

Det. Temp:
250°C

Det. Type:
MSD



5Ex085
86
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Manufactured By: MEW

John Lidgett

John Lidgett - QA Analyst

1 Expiration date of the unopened ampul stored at recommended temperature.
2 Purity was determined by one or more of the following techniques: GC/FID, HPLC, GC/ECD, GC/MS. Value rounded to the nearest LOWER whole percentage. In addtion to detectors listed above, chemical identity and purity are confirmed using 1 or more of the following: MS, DSC, solid probe MS, GC/FPD, GC/NPD, GC/TC, FTIR, melting point, reftactive index, and Karl Fisher. See data pack or contact Restek for further details.
3 Based upon gravimetric preperation with balance calibration verified using NISTtraceable weights (7 mass levels).
4 Percent Uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.

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110 Benner Circle
Bellefonte, PA 16823-8812
Tel: (800) 356-1688
Fax: (814) 353-1309

Catalog No.: 30499 **Lot No.:** A037729

Description: Acrolein Standard

Expiration Date[1]: October 2005 **Storage:** Freezer

Elution Order	Compound	CAS#	Percent Purity[2]	Concentration[3]	Percent Uncertainty[4]
1	Acrolein	107-02-8	99%	10000 ug/mL	+/- 0.1
	Solvent: P&T Methanol	67-56-1	99%		

Column:
105m x .53mm x 3.0um
Rtx-502.2 (cat.#10910)

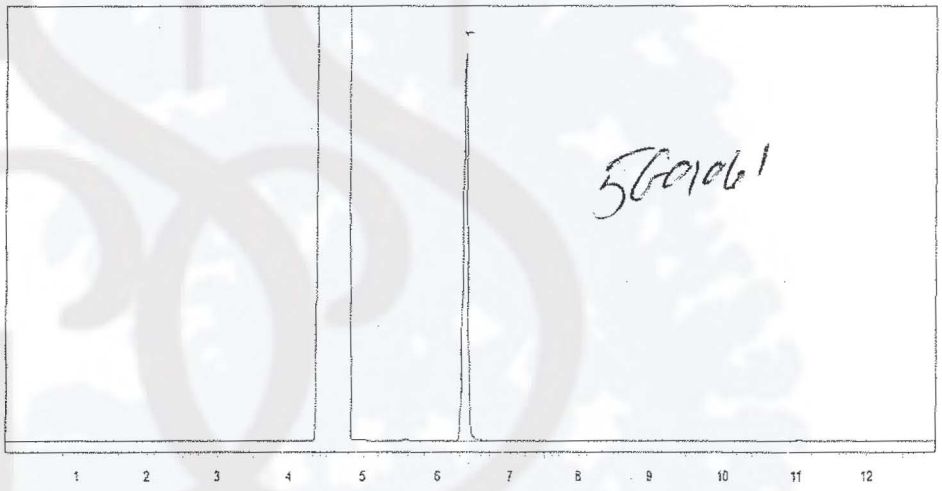
Carrier Gas:
hydrogen @ 40 cm/sec

Temp. Program:
40°C (hold 2 min.) to 200°C
@ 8°C/min.

Inj. Temp:
200°C

Det. Temp:
250°C

Det. Type:
FID



Manufactured By: n/a

John Lidgett - QA Analyst

1 Expiration date of the unopened ampul stored at recommended temperature.
2 Purity was determined by one or more of the following techniques: GC/FID, HPLC, GC/ECD, GC/MS. Value rounded to the nearest LOWER whole percentage. In addtion to detectors listed above, chemical identity and purity are confirmed using 1 or more of the following: MS, DSC, solid probe MS, GC/FPD, GC/NPD, GC/TC, FTIR, melting point, reftactive index, and Karl Fisher. See data pack or contact Restek for further details.
3 Based upon gravimetric preperation with balance calibration verified using NISTtraceable weights (7 mass levels).
4 Percent Uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.

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Internal Standard Mixture

Product STM-520 **Expiration Date:** Jan-2008
Lot Number: CA-2291 **Page:** 1 of 1

This Certified Reference Material (CRM) was manufactured and verified in accordance with ULTRA's ISO 9001:2000 registered quality system, and the analyte concentrations were verified by our ISO 17025 accredited laboratory. The true value and uncertainty value at the 95% confidence level for each analyte, determined gravimetrically, is listed below.

Analyte	CAS#	Analyte Lot	True Value
chlorobenzene-d5	003114-55-4	PR-12098	2509 ± 13 µg/mL
1,4-dichlorobenzene-d4	003855-82-1	16304CA	2503 ± 13 µg/mL
fluorobenzene	000462-06-6	CS10731MG	2507 ± 13 µg/mL

Matrix: methanol (methyl alcohol)

5H01052
53
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Balances used in the manufacture of this standard are calibrated with weights traceable to NIST in compliance with ANSI/NCSL Z-540-1 and ISO 9001.

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Edward Fitzgerald
Dr. Edward Fitzgerald, Senior Scientist

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Certificate of Analysis

Method 8260 Surrogate Standard Mixture

Product STM-530 **Expiration Date:** Jul-2008
Lot Number: CB-1395 **Page:** 1 of 1

This Certified Reference Material (CRM) was manufactured and verified in accordance with ULTRA's ISO 9001:2000 registered quality system, and the analyte concentrations were verified by our ISO 17025 accredited laboratory. The true value and uncertainty value at the 95% confidence level for each analyte, determined gravimetrically, is listed below.

Analyte	CAS#	Analyte Lot	True Value
4-bromofluorobenzene	000460-00-4	11203KS	2503 ± 13 µg/mL
dibromofluoromethane	001868-53-7	90004843	2506 ± 13 µg/mL
1,2-dichloroethane-d4	017060-07-0	PSO2F-623	2508 ± 13 µg/mL
toluene-d8	002037-26-5	PSOAG-433	2506 ± 13 µg/mL

Matrix: methanol (methyl alcohol)

5 H0106
62
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68

Balances used in the manufacture of this standard are calibrated with weights traceable to NIST in compliance with ANSI/NCSL Z-540-1 and ISO 9001.

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Edward Fitzgerald
Dr. Edward Fitzgerald,
Senior Scientist

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Custom Standard

Product Number: CUS-6492 **Expiration Date:** Apr-2007

Lot Number: CB-0748 **Page:** 1 of 1

This Certified Reference Material (CRM) was manufactured and verified in accordance with ULTRA's ISO 9001:2000 registered quality system. The true value and uncertainty value at the 95% confidence level for each analyte, determined gravimetrically, is listed below.

Analyte	CAS#	Analyte Lot	True Value
diisopropyl ether	000108-20-3	02151KQ	2005 ± 10 µg/mL
ethyl tert-butyl ether	000637-92-3	MN-17917	2006 ± 10 µg/mL
TAME	000994-05-8	JN-12512EN	2005 ± 10 µg/mL
1-chlorohexane	000544-10-5	232-8B	2008 ± 10 µg/mL
cyclohexane	000110-82-7	380707	2005 ± 10 µg/mL
methyl acetate	000079-20-9	I-136A	2004 ± 10 µg/mL
methylcyclohexane	000108-87-2	DR-00844CR	2005 ± 10 µg/mL
1,1,2-trichlorotrifluoroethane	000076-13-1	KN-09246KN	2005 ± 10 µg/mL

Matrix: methanol (methyl alcohol)

5 Pos 60
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66 67

MASTER

Balances used in the manufacture of this standard are calibrated with weights traceable to NIST in compliance with ANSI/NCSL Z-540-1 and ISO 9001.

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Dr. Edward Fitzgerald,
Senior Scientist

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VOC Mixture

Product Number: DWM-592 **Expiration Date:** May-2007

Lot Number: W-0239 **Page:** 1 of 2

This Certified Reference Material (CRM) was manufactured and verified in accordance with ULTRA's ISO 9001:2000 registered quality system, and the analyte concentrations were verified by our ISO 17025 accredited laboratory. The true value and uncertainty value at the 95% confidence level for each analyte, determined gravimetrically, is listed below.

Analyte	CAS#	Analyte Lot	True Value
acetone	000067-64-1	43034	2008 ± 10 µg/mL
acrylonitrile	000107-13-1	HI11505EI	2009 ± 10 µg/mL
allyl chloride	000107-05-1	MC070777	2009 ± 10 µg/mL
2-butanone	000078-93-3	CA-02161PO	2010 ± 10 µg/mL
carbon disulfide	000075-15-0	AI12076LU	2008 ± 10 µg/mL
chloroacetonitrile	000107-14-2	CF01026AF	2005 ± 10 µg/mL
1-chlorobutane	000109-69-3	07247HF	2007 ± 10 µg/mL
trans-1,4-dichloro-2-butene	000110-57-6	JN01422DG	2008 ± 10 µg/mL
1,1-dichloro-2-propanone	000513-88-2	LY02111DL	2007 ± 10 µg/mL
diethyl ether	000060-29-7	MA01256EA	2008 ± 10 µg/mL
ethyl methacrylate	000097-63-2	AO09819AS	2008 ± 10 µg/mL
hexachloroethane	000067-72-1	DN06203HF	2005 ± 10 µg/mL
2-hexanone	000591-78-6	AI11228KS	2009 ± 10 µg/mL
methacrylonitrile	000126-98-7	LT00427ET	2009 ± 10 µg/mL
methyl acrylate	000096-33-3	HF01125BF	2008 ± 10 µg/mL
methyl iodide	000074-88-4	KA20329DA	2008 ± 10 µg/mL
methyl methacrylate	000080-62-6	HN03906DN	2007 ± 10 µg/mL
4-methyl-2-pentanone	000108-10-1	05756LQ	2007 ± 10 µg/mL
tert-butylmethyl ether	001634-04-4	36032	2009 ± 10 µg/mL
nitrobenzene	000098-95-3	HK2925LK	2008 ± 10 µg/mL
2-nitropropane	000079-46-9	BZ01727PY	2004 ± 10 µg/mL

Balances used in the manufacture of this standard are calibrated with weights traceable to NIST in compliance with ANSI/NCSL Z-540-1 and ISO 9001.

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Certificate of Analysis

VOC Mixture

Product Number: DWM-592
Lot Number: W-0239
Expiration Date: May-2007
Page: 2 of 2

Analyte	CAS#	Analyte Lot	True Value
pentachloroethane	000076-01-7	232-36B	2007 ± 10 µg/mL
propionitrile	000107-12-0	BI18125KS	2008 ± 10 µg/mL
tetrahydrofuran	000109-99-9	EN02837AN	2008 ± 10 µg/mL

Matrix: methanol (methyl alcohol)

Balances used in the manufacture of this standard are calibrated with weights traceable to NIST in compliance with ANSI/NCSL Z-540-1 and ISO 9001.

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VOC Mixture

Product Number: DWM-588 **Expiration Date:** Apr-2008

Lot Number: CB-0526 **Page:** 1 of 3

This Certified Reference Material (CRM) was manufactured and verified in accordance with ULTRA's ISO 9001:2000 registered quality system, and the analyte concentrations were verified by our ISO 17025 accredited laboratory. The true value and uncertainty value at the 95% confidence level for each analyte, determined gravimetrically, is listed below.

Analyte	CAS#	Analyte Lot	True Value
bromochloromethane	000074-97-5	JS-16015HS	2006 ± 10 µg/mL
bromodichloromethane	000075-27-4	DU-14522LS	2006 ± 10 µg/mL
bromoform	000075-25-2	DU-06126KS	2004 ± 10 µg/mL
carbon tetrachloride	000056-23-5	01704MF	2006 ± 10 µg/mL
chloroform	000067-66-3	BS-03041BS	2005 ± 10 µg/mL
dibromochloromethane	000124-48-1	DO-12622CI	2006 ± 10 µg/mL
dibromomethane	000074-95-3	EM-01514TJ	2005 ± 10 µg/mL
methylene chloride	000075-09-2	43041	2006 ± 10 µg/mL
trichlorofluoromethane	000075-69-4	DR-16417BR	2007 ± 10 µg/mL
1,2-dibromoethane	000106-93-4	TB-101777	2006 ± 10 µg/mL
1,1-dichloroethane	000075-34-3	64552/1	2005 ± 10 µg/mL
1,2-dichloroethane	000107-06-2	KN-09446KN	2006 ± 10 µg/mL
1,1-dichloroethene	000075-35-4	CA-02207CA	2005 ± 10 µg/mL
cis-1,2-dichloroethene	000156-59-2	13707BO	2006 ± 10 µg/mL
trans-1,2-dichloroethene	000156-60-5	DO-07817JR	2006 ± 10 µg/mL
1,1,1,2-tetrachloroethane	000630-20-6	CO-12312LI	2005 ± 10 µg/mL
1,1,2,2-tetrachloroethane	000079-34-5	10917TB	2006 ± 10 µg/mL
tetrachloroethene	000127-18-4	PS-00344BR	2006 ± 10 µg/mL
1,1,1-trichloroethane	000071-55-6	LU-13149TR	2006 ± 10 µg/mL
1,1,2-trichloroethane	000079-00-5	JB-0701HH	2006 ± 10 µg/mL
trichloroethene	000079-01-6	KN-08846KN	2005 ± 10 µg/mL

5E20094
95
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Balances used in the manufacture of this standard are calibrated with weights traceable to NIST in compliance with ANSI/NCSL Z-540-1 and ISO 9001.

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Product Number: DWM-588 **Expiration Date:** Apr-2008

Lot Number: CB-0526 **Page:** 2 of 3

Analyte	CAS#	Analyte Lot	True Value
1,2-dibromo-3-chloropropane	000096-12-8	OGF-01	2006 ± 10 µg/mL
1,2-dichloropropane	000078-87-5	DC-120777	2005 ± 10 µg/mL
1,3-dichloropropane	000142-28-9	PR-17916MR	2006 ± 10 µg/mL
2,2-dichloropropane	000594-20-7	CI-05304BI	2006 ± 10 µg/mL
1,1-dichloropropene	000563-58-6	34768-21	2006 ± 10 µg/mL
cis-1,3-dichloropropene	010061-01-5	35072-03	2007 ± 10 µg/mL
trans-1,3-dichloropropene	010061-02-6	34257-41	2005 ± 10 µg/mL
hexachlorobutadiene	000087-68-3	PB-082017	2006 ± 10 µg/mL
1,2,3-trichloropropane	000096-18-4	EY-01328EY	2006 ± 10 µg/mL
naphthalene	000091-20-3	N960012	2006 ± 10 µg/mL
benzene	000071-43-2	31072	2005 ± 10 µg/mL
n-butylbenzene	000104-51-8	AA-28519CO	2006 ± 10 µg/mL
sec-butylbenzene	000135-98-8	MR-11305DN	2006 ± 10 µg/mL
tert-butylbenzene	000098-06-6	MQ-04010MQ	2005 ± 10 µg/mL
ethylbenzene	000100-41-4	033067	2006 ± 10 µg/mL
isopropylbenzene	000098-82-8	EN-00621TG	2006 ± 10 µg/mL
4-isopropyltoluene	000099-87-6	PP-05104CP	2006 ± 10 µg/mL
n-propylbenzene	000103-65-1	LO-14503MR	2005 ± 10 µg/mL
styrene	000100-42-5	MQ-11228MQ	2005 ± 10 µg/mL
toluene	000108-88-3	34117	2006 ± 10 µg/mL
1,2,4-trimethylbenzene	000095-63-6	BO-13528BI	2006 ± 10 µg/mL
1,3,5-trimethylbenzene	000108-67-8	KM-02011HM	2005 ± 10 µg/mL
o-xylene	000095-47-6	DO-06834CO	2006 ± 10 µg/mL
m-xylene	000108-38-3	DI-00459CI	2005 ± 10 µg/mL

Balances used in the manufacture of this standard are calibrated with weights traceable to NIST in compliance with ANSI/NCSL Z-540-1 and ISO 9001.

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Product Number: DWM-588 **Expiration Date:** Apr-2008

Lot Number: CB-0526 **Page:** 3 of 3

Analyte	CAS#	Analyte Lot	True Value
p-xylene	000106-42-3	03747LN	2005 ± 10 µg/mL
1,4-dichlorobenzene	000106-46-7	06205KA	2004 ± 10 µg/mL
bromobenzene	000108-86-1	CG-02513MF	2006 ± 10 µg/mL
chlorobenzene	000108-90-7	63148HZ	2006 ± 10 µg/mL
2-chlorotoluene	000095-49-8	KS-06506BN	2006 ± 10 µg/mL
4-chlorotoluene	000106-43-4	CR-14512LQ	2005 ± 10 µg/mL
1,2-dichlorobenzene	000095-50-1	08946KY	2005 ± 10 µg/mL
1,3-dichlorobenzene	000541-73-1	JN-05902LZ	2006 ± 10 µg/mL
1,2,3-trichlorobenzene	000087-61-6	LI-12912PF	2005 ± 10 µg/mL
1,2,4-trichlorobenzene	000120-82-1	00334TQ	2006 ± 10 µg/mL
bromomethane	000074-83-9	06623AQ	2008 ± 10 µg/mL
chloroethane	000075-00-3	00223KG	2008 ± 10 µg/mL
chloromethane	000074-87-3	07-44048	2008 ± 10 µg/mL
dichlorodifluoromethane	000075-71-8	N960053	2008 ± 10 µg/mL
vinyl chloride	000075-01-4	UN-1086	2009 ± 10 µg/mL

Matrix: methanol (methyl alcohol)

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Balances used in the manufacture of this standard are calibrated with weights traceable to NIST in compliance with ANSI/NCSL Z-540-1 and ISO 9001.

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AccuStandard Inc.
125 Market Street
New Haven, CT 06513
USA

CERTIFICATE OF ANALYSIS

Ph: 203-786-5290
Fax: 203-786-5287
E-mail: usa@accustandard.com
www.accustandard.com

CATALOG NO. M-601C-10X
DESCRIPTION: 2-Chloroethylvinyl ether
LOT: B4100153
SOLVENT: MeOH

EXPIRATION: Oct 20, 2007

The uncertainty in the preparation of this standard is <5% if the concentration is less than or equal to 200µg/mL. For concentrations greater than 200µg/mL the uncertainty is <2%.

Component	CAS #	Purity % (GC/MS)	Gravimetric Concentration[1] (µg/mL)	Analyte Concentration[2] (µg/mL)
2-Chloroethylvinyl ether	110-75-8	99.7	2000	1994

5E20098
99
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101

Please note: AccuStandard follows the U.S. conventions in reporting numerical values, on both certificates and labels.

A comma (,) is used to separate units of one-thousand or greater.
A period (.) is used as a decimal place marker.

1. All weights are traceable through National Institute of Standards & Technology, Test No. 822/254480.
2. Analyte Concentration = Purity x Gravimetric Concentration
3. A product with a suffix (-1A, -2B, etc.) on its lot# has had its expiration date extended and is identical to the same lot# without the suffix.

Certified by: R. Cooper

This product was manufactured to meet the quality system requirements of ISO 9001

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AccuStandard Inc.
125 Market Street
New Haven, CT 06513
USA

Ph: 203-786-5290
Fax: 203-786-5287
E-mail: usa@accustandard.com
www.accustandard.com

CERTIFICATE OF ANALYSIS

CATALOG NO. APP-9-007-10X
DESCRIPTION: Acrolein
LOT: B5060041
SOLVENT: MeOH

EXPIRATION: Oct 5, 2005

The uncertainty in the preparation of this standard is <5% if the concentration is less than or equal to 200µg/mL. For concentrations greater than 200µg/mL the uncertainty is <2%.

Component	CAS #	Purity % MFG	Gravimetric Concentration[1] (µg/mL)	Analyte Concentration[2] (µg/mL)
Acrolein	107-02-8	90.0	1112*	1001

560638
39

* Weight compensated to 100% purity

1. All weights are traceable through National Institute of Standards & Technology, Test No. 822/254480
2. Analyte Concentration = Purity x Gravimetric Concentration
3. A product with a suffix (-1A, -2B, etc.) on its lot# has had its expiration date extended and is identical to the same lot# without the suffix.

Please note: AccuStandard follows the U.S. conventions in reporting numerical values, on both certificates and labels.

A comma (,) is used to separate units of one-thousand or greater.
A period (.) is used as a decimal place marker.

Certified by: R. Cooper

This product was manufactured to meet the quality system requirements of ISO 9001

QR-ORG/INO-001
Rev. 11/02

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AccuStandard Inc.
125 Market Street
New Haven, CT 06513
USA

Ph: 203-786-5290
Fax: 203-786-5287
E-mail: usa@accustandard.com
www.accustandard.com

CERTIFICATE OF ANALYSIS

CATALOG NO. APP-9-211-20X
DESCRIPTION: Vinyl acetate
LOT: B5020138-1A
SOLVENT: MeOH

EXPIRATION: Nov 20, 2005

The uncertainty in the preparation of this standard is <5% if the concentration is less than or equal to 200µg/mL. For concentrations greater than 200µg/mL the uncertainty is <2%.

Component	CAS #	Purity % (GC/FID)	Gravimetric Concentration[1] (µg/mL)	Analyte Concentration[2] (µg/mL)
Vinyl acetate	108-05-4	99.9	2001	1999

5/06/04
41

ACCUSTANDARD LABORATORY
MASTER

1. All weights are traceable through National Institute of Standards & Technology, Test No. 822/254480
2. Analyte Concentration = Purity x Gravimetric Concentration
3. A product with a suffix (-1A, -2B, etc.) on its lot# has had its expiration date extended and is identical to the same lot# without the suffix.

Please note: AccuStandard follows the U.S. conventions in reporting numerical values, on both certificates and labels.

A comma (,) is used to separate units of one-thousand or greater.
A period (.) is used as a decimal place marker.

Certified by: R. Cooper

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ULTRA SCIENTIFIC
Analytical Solutions

Certificate of Analysis

1,4-Dioxane

Product Number: RCC-180 **Expiration Date:** Jan-2008

Lot Number: NT02237 **Page:** 1 of 1

This reference material has been analyzed by high resolution gas chromatography or high performance liquid chromatography, and found to meet the specifications stated below. The uncertainty of the purity measurement is ± 0.5%.

Compound	CAS #	Purity
1,4-dioxane	000123-91-1	99%

Storage: May be stored at room temperature

5G-11049

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Quality System
Quality Endorsed Company
ISO 9001
SAI Global Registered

ACCREDITED
ISO 17025
Cert. No. 0851-01

250 Smith Street, North Kingstown, RI 02852 USA
401-294-9400 Fax: 401-295-2330
www.ultrasci.com

Edward Fitzgerald

Dr. Edward Fitzgerald,
Senior Scientist

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Certificate of Analysis

DESCRIPTION: 1,4-Dioxane

MFG. DATE: Nov 2004

CATALOG NO.: 442251 (1)

LOT NO.: LB25729

EXP. DATE: Nov 2007

CAS NUMBER: 123-91-1

MOLECULAR FORMULA: C4H8O2

MOLECULAR WEIGHT: 88.1

PHYSICAL PROPERTIES	ASSAY	
FTIR	Matches: SEA 207	Lib. No.: 1563
GC - Mass Spec	Matches: NIST	Lib. No.: 62899
Purity (2)	99.9%	
Refractive index	1.4213 @ 20.0 deg C	

5C0961

(1) This product is packaged from R472225 Lot number LB11219.

(2) Determined by GC-FID unless otherwise noted.

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Elwood Doughty

Elwood Doughty
Quality Control Supervisor

Supelco warrants that its products conform to the information contained in this publication. Purchaser must determine the suitability of the product for its particular use. Please see the latest catalog or order invoice and packing slip for additional terms and conditions of sale.

SUPELCO
595 North Harrison Road
Bellefonte, PA 16823-0048 USA
Phone (814)359-3441

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TOPLEVEL PARAMETERS

Method Information For: C:\HPCHEM\1\METHODS\AQ080505.M

Method Sections To Run:

( ) Save Copy of Method With Data
( ) Pre-Run Cmd/Macro =
(X) Data Acquisition
(X) Data Analysis
( ) Post-Run Cmd/Macro =

MS-3

Method Comments:
Method 8260

END OF TOPLEVEL PARAMETERS

INSTRUMENT CONTROL PARAMETERS

Sample Inlet: GC
Injection Source: Manual
Injection Location: Rear
Mass Spectrometer: Enabled

HP5890 Temperature Parameters

Zone Temperatures:	State	Setpoint
Inlet A:	Off	50 C
Inlet B:	On	200 C
Detector A:	Off	50 C
Detector B:	On	280 C
Auxiliary:	Off	50 C

Oven Parameters:	
Oven Equib Time:	0.20 minutes
Oven Max:	260 C
Oven State:	On
Cryo State:	Off
Cryo Blast:	Off
Ambient:	25 C

Oven Program:

Initial Temperature:	40 C
Initial Time:	4.00 minutes

Level	Rate (C/minute)	Final Temperature (C)	Final Time (minutes)
1	8.0	150	2.00
2(A)	35.0	220	4.25
3(B)	0.0	0	0.00
Next Run Time:		26.00 minutes	

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HP5890 Inlet Pressure Programs

GC Pressure Units: psi

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Inlet A:

Constant Flow:	Off
Constant Flow Pressure:	0.0 psi
Constant Flow Temperature:	50 C
Initial Pressure:	0.0 psi
Initial Time:	650.00 minutes

Level	Rate (psi/minute)	Final Pressure (psi)	Final Time (minutes)
1	0.00	0.0	0.00
2(A)	0.00	0.0	0.00
3(B)	0.00	0.0	0.00

Total Program Time: 650.00 minutes

Column Length:	30.00 m
Column Diameter:	0.530 mm
Gas:	He
Vacuum Compensation:	Off

Inlet B:

Constant Flow:	On
Constant Flow Pressure:	19.4 psi
Constant Flow Temperature:	40 C
Initial Pressure:	0.0 psi
Initial Time:	650.00 minutes

Level	Rate (psi/minute)	Final Pressure (psi)	Final Time (minutes)
1	0.00	0.0	0.00
2(A)	0.00	0.0	0.00
3(B)	0.00	0.0	0.00

Total Program Time: 650.00 minutes

Column Length:	60.00 m
Column Diameter:	0.250 mm
Gas:	He
Vacuum Compensation:	Off

HP5890 Inlet Split Flow Control

Channel C:
Carrier gas supply for channel C is not linked to a split vent.

Channel D:
Carrier gas supply for channel D is not linked to a split vent.

Channel E:
Carrier gas supply for channel E is not linked to a split vent.

Channel F:
Carrier gas supply for channel F is not linked to a split vent.

HP5890 Packed Column Flow Control

Inlet A not used to control packed column flow.

Inlet B not used to control packed column flow.

HP5890 Auxiliary Pressure Programs

Channel C:
Comment:

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Pressure Program:
Initial Pressure: 0.0 psi
Initial Time: 650.00 minutes

Level	Rate (psi/minute)	Final Pressure (psi)	Final Time (minutes)
1	0.00	0.0	0.00
2(A)	0.00	0.0	0.00
3(B)	0.00	0.0	0.00
Total Program Time:		650.00 minutes	

Make-up Gas Compensation: None

Channel D:
Comment:

Pressure Program:
Initial Pressure: 0.0 psi
Initial Time: 650.00 minutes

Level	Rate (psi/minute)	Final Pressure (psi)	Final Time (minutes)
1	0.00	0.0	0.00
2(A)	0.00	0.0	0.00
3(B)	0.00	0.0	0.00
Total Program Time:		650.00 minutes	

Make-up Gas Compensation: None

Channel E:
Comment:

Pressure Program:
Initial Pressure: 0.0 psi
Initial Time: 650.00 minutes

Level	Rate (psi/minute)	Final Pressure (psi)	Final Time (minutes)
1	0.00	0.0	0.00
2(A)	0.00	0.0	0.00
3(B)	0.00	0.0	0.00
Total Program Time:		650.00 minutes	

Make-up Gas Compensation: None

Channel F:
Comment:

Pressure Program:
Initial Pressure: 0.0 psi
Initial Time: 650.00 minutes

Level	Rate (psi/minute)	Final Pressure (psi)	Final Time (minutes)
1	0.00	0.0	0.00
2(A)	0.00	0.0	0.00
3(B)	0.00	0.0	0.00
Total Program Time:		650.00 minutes	

Make-up Gas Compensation: None

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HP5890 Purge Valve Settings

Inlet Purge	Init Value	On Time	Off Time	Splitless Injection
A	Off	0.00	0.00	No
B	On	0.00	0.00	No

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HP5890 Valve and Relay Information

Initial Setpoints:

5890 Valves:

Valve 1: Off	Valve 2: Off	Valve 3: Off	Valve 4: On

19405 Valves:

Valve 5: Off	Valve 6: Off	Valve 7: Off	Valve 8: Off

19405 Relays:

Relay 1: Off	Relay 2: Off	Relay 3: Off	Relay 4: Off

HP5890 Detector Information

Detector	Type	State
A	---	Off
B	---	Off

HP5890 Signal Information

Not saving signal data.

Signal	Source	Peak Width	Data Rate	Start Data	Stop Data
1	Testplot	0.053	5.000	0.00	1.00
2	Testplot	0.053	5.000	0.00	1.00

MS ACQUISITION PARAMETERS

General Information
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Tune File : BFB.U
Acquistion Mode : Scan

MS Information
-- -----------

Solvent Delay : 3.70 min

EM Absolute : True
Resulting EM Voltage : 1941.2

[Scan Parameters]

Low Mass : 35
High Mass : 260
Threshold : 800
Sample # : 4 A/D Samples 16
Plot 2 low mass : 50
Plot 2 high mass : 550

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END OF MS ACQUISITION PARAMETERS

END OF INSTRUMENT CONTROL PARAMETERS

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DATA ANALYSIS PARAMETERS

Method Name: C:\HPCHEM\1\METHODS\AQ080505.M

Percent Report Settings

Sort By: Signal

Output Destination
    Screen: No
    Printer: Yes
    File: No

Integration Events: Meth Default

Generate Report During Run Method: No

Signal Correlation Window: 0.020

Qualitative Report Settings

Peak Location of Unknown: Apex

Library to Search	Minimum Quality
c:\database\nbs75k.l	0

Integration Events: Meth Default

Report Type: Summary

Output Destination
    Screen: No
    Printer: Yes
    File: No

Generate Report During Run Method: No

Quantitative Report Settings

Report Type: Summary

Output Destination
    Screen: No
    Printer: No
    File: No

Generate Report During Run Method: Yes

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ELEMENT ID: 0508010
Calibration Last Updated: Tue Aug 30 15:15:34 2005

Reference Window: 10.00 Percent
Non-Reference Window: 5.00 Percent

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Correlation Window: 0.02 minutes
Default Multiplier: 1.00
Default Sample Concentration: 0.00

Compound Information

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1) Fluorobenzene (ISTD)

Ret. Time 12.61 min., Extract & Integrate from 12.11 to 13.11 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 96.00			*** METH DEFAULT ***
Q1 70.00	16.80	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	1793239
10		25.000	1801571
5		25.000	1838338
1		25.000	1704091
50		25.000	1818145
100		25.000	1899842
0.5		25.000	1716216

Qualifier Peak Analysis ON ISTD conc: 25.000 ug/l
Curve Fit: Avg. RF

---

2) Dichlorodifluoromethane ( )

Ret. Time 4.15 min., Extract & Integrate from 3.65 to 4.65 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 85.00			*** METH DEFAULT ***
Q1 87.00	33.20	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	416355
10		10.000	172274
5		5.000	90302
1		1.000	18792
50		50.000	836422
100		100.000	1823963
0.5	not used for this compound		

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

3) Chloromethane ( )

Ret. Time 4.46 min., Extract & Integrate from 3.96 to 4.96 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 50.00			*** METH DEFAULT ***
Q1 52.00	33.10	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	597482
10		10.000	266628
5		5.000	137597
1		1.000	30884
50		50.000	1203850
100		100.000	2480172
0.5	not used for this compound		

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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4) Vinyl Chloride ( )

Ret. Time 4.79 min., Extract & Integrate from 4.29 to 5.29 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 62.00			*** METH DEFAULT ***
Q1 64.00	38.10	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	437518
10		10.000	192044
5		5.000	101731
1		1.000	19224
50		50.000	882458
100		100.000	1837800
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

5) Bromomethane ( )

Ret. Time 5.47 min., Extract & Integrate from 4.97 to 5.97 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 94.00			*** METH DEFAULT ***
Q1 96.00	95.80	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	301032
10		10.000	135055
5		5.000	77283
1		1.000	18213
50		50.000	649844
100		100.000	1436601
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Linear

---

6) Chloroethane ( )

Ret. Time 5.72 min., Extract & Integrate from 5.22 to 6.22 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 64.00			*** METH DEFAULT ***
Q1 66.00	32.00	30.0	*** METH DEFAULT ***
Q2 49.00	32.40	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	256692
10		10.000	112808
5		5.000	57979
1		1.000	16574
50		50.000	495501
100		100.000	1066476
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Linear

---

7) Trichlorofluoromethane ( )

Ret. Time 6.68 min., Extract & Integrate from 6.18 to 7.18 min.

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Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 101.00			*** METH DEFAULT ***
Q1 103.00	67.50	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
25	25.000	399557
10	10.000	165001
5	5.000	81556
1	1.000	19818
50	50.000	799662
100	100.000	1678945
0.5	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

8) Diethyl ether ( )

Ret. Time 7.09 min., Extract & Integrate from 6.59 to 7.59 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 59.00			*** METH DEFAULT ***
Q1 45.00	102.20	30.0	*** METH DEFAULT ***
Q2 74.00	63.70	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
25	25.000	220431
10	10.000	102527
5	5.000	52949
1	1.000	18591
50	50.000	468962
100	100.000	1051883
0.5	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Linear

---

9) Acrolein ( )

Ret. Time 6.68 min., Extract & Integrate from 6.18 to 7.18 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 56.00			*** METH DEFAULT ***
Q1 55.00	71.60	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
25	25.000	42726
10	10.000	18902
5	5.000	-1
1	1.000	-1
50	50.000	72056
100	100.000	165907
0.5	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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10) Acetone ( )

Ret. Time 6.88 min., Extract & Integrate from 6.38 to 7.38 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 58.00			*** METH DEFAULT ***
Q1 43.00	388.40	30.0	*** METH DEFAULT ***

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Lvl	ID	Conc (ug/l)	Response
25		125.000	99057
10		50.000	47222
5		25.000	30787
1		5.000	-1
50		250.000	176366
100		500.000	382429
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Linear

---

11) Iodomethane ( )

Ret. Time 7.60 min., Extract & Integrate from 7.10 to 8.10 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	142.00			*** METH DEFAULT ***
Q1	126.90	54.90	30.0	*** METH DEFAULT ***
Q2	141.00	15.60	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	500804
10		10.000	206208
5		5.000	106626
1		1.000	31182
50		50.000	1120745
100		100.000	2489139
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Linear

---

12) 1,1,2-Trichloro-1,2,2-trifluoroethane ( )

Ret. Time 7.88 min., Extract & Integrate from 7.38 to 8.38 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	101.00			*** METH DEFAULT ***
Q1	151.00	59.40	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	331875
10		10.000	145444
5		5.000	87959
1		1.000	16874
50		50.000	695665
100		100.000	1381718
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

13) Methyl Acetate ( )

Ret. Time 7.90 min., Extract & Integrate from 7.40 to 8.40 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	43.00			*** METH DEFAULT ***
Q1	74.00	14.90	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	226125
10		10.000	108222
5		5.000	60912
1		1.000	-1

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50	50.000	467109
100	100.000	960526
0.5	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

14) Allyl Chloride ( )

Ret. Time 7.94 min., Extract & Integrate from 7.44 to 8.44 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 41.00			*** METH DEFAULT ***
Q1 76.00	25.20	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	671399
10		10.000	288531
5		5.000	162257
1		1.000	36376
50		50.000	1561946
100		100.000	3343008
0.5	not used for this compound		

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

15) Carbon Disulfide ( )

Ret. Time 8.12 min., Extract & Integrate from 7.62 to 8.62 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 76.00			*** METH DEFAULT ***
Q1 78.00	9.40	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	1537785
10		10.000	650059
5		5.000	331521
1		1.000	70014
50		50.000	3411160
100		100.000	7456603
0.5	not used for this compound		

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

16) 1,1-Dichloroethene ( )

Ret. Time 7.55 min., Extract & Integrate from 7.05 to 8.05 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 96.00			*** METH DEFAULT ***
Q1 61.00	114.60	30.0	*** METH DEFAULT ***
Q2 63.00	37.30	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	300278
10		10.000	128316
5		5.000	62540
1		1.000	20157
50		50.000	621349
100		100.000	1302793
0.5	not used for this compound		

Qualifier Peak Analysis ON

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Curve Fit: Linear

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17) Methylene Chloride ( )

Ret. Time 7.79 min., Extract & Integrate from 7.29 to 8.29 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	84.00			*** METH DEFAULT ***
Q1	86.00	64.60	30.0	*** METH DEFAULT ***
Q2	49.00	147.10	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	359532
10		10.000	154815
5		5.000	83431
1		1.000	23476
50		50.000	726794
100		100.000	1554380
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Linear

---

18) Methyl tert-Butyl Ether ( )

Ret. Time 9.06 min., Extract & Integrate from 8.56 to 9.56 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	73.00			*** METH DEFAULT ***
Q1	57.00	25.90	30.0	*** METH DEFAULT ***
Q2	41.00	29.90	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	800869
10		10.000	335324
5		5.000	149753
1		1.000	31545
50		50.000	1817014
100		100.000	4152248
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

19) Acrylonitrile ( )

Ret. Time 7.66 min., Extract & Integrate from 7.16 to 8.16 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	53.00			*** METH DEFAULT ***
Q1	52.00	82.60	30.0	*** METH DEFAULT ***
Q2	51.00	38.40	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	92809
10		10.000	44811
5		5.000	23381
1		1.000	-1
50		50.000	190253
100		100.000	405352
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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20) trans-1,2-Dichloroethene ( )

Ret. Time 8.86 min., Extract & Integrate from 8.36 to 9.36 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	96.00			*** METH DEFAULT ***
Q1	61.00	113.30	30.0	*** METH DEFAULT ***
Q2	98.00	66.50	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	473996
10		10.000	200838
5		5.000	97241
1		1.000	18775
50		50.000	1052229
100		100.000	2340829
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

21) 1,1-Dichloroethane ( )

Ret. Time 9.25 min., Extract & Integrate from 8.75 to 9.75 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	63.00			*** METH DEFAULT ***
Q1	65.00	32.60	30.0	*** METH DEFAULT ***
Q2	83.00	14.40	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	1084415
10		10.000	469088
5		5.000	228337
1		1.000	50777
50		50.000	2235019
100		100.000	4726697
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

22) Vinyl Acetate ( )

Ret. Time 9.49 min., Extract & Integrate from 8.99 to 9.99 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	43.00			*** METH DEFAULT ***
Q1	86.00	7.10	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	969560
10		10.000	398756
5		5.000	192408
1		1.000	39563
50		50.000	2294813
100		100.000	5426019
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

23) Chloroprene ( )

Ret. Time 9.83 min., Extract & Integrate from 9.33 to 10.33 min.

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Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 53.00			*** METH DEFAULT ***
Q1 88.00	66.80	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	730206
10		10.000	278040
5		5.000	133241
1		1.000	27007
50		50.000	1598316
100		100.000	3503002
0.5	not used for this compound		

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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24) 2-Butanone ( )

Ret. Time 9.95 min., Extract & Integrate from 9.45 to 10.45 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 72.00			*** METH DEFAULT ***
Q1 43.00	695.40	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		125.000	143841
10		50.000	62943
5		25.000	33898
1		5.000	-1
50		250.000	307714
100		500.000	668126
0.5	not used for this compound		

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

25) Di-isopropyl ether ( )

Ret. Time 9.98 min., Extract & Integrate from 9.48 to 10.48 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 45.00			*** METH DEFAULT ***
Q1 43.00	48.10	30.0	*** METH DEFAULT ***
Q2 87.00	16.50	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	2337668
10		10.000	942710
5		5.000	426010
1		1.000	76440
50		50.000	4992657
100		100.000	10505860
0.5	not used for this compound		

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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26) Methacrylonitrile ( )

Ret. Time 10.10 min., Extract & Integrate from 9.60 to 10.60 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 41.00			*** METH DEFAULT ***
Q1 67.00	42.30	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response

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25	25.000	281255
10	10.000	123283
5	5.000	56666
1	1.000	8378
50	50.000	615653
100	100.000	1310989
0.5	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

27) cis-1,2 Dichloroethene ( )

Ret. Time 10.16 min., Extract & Integrate from 9.66 to 10.66 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 96.00			*** METH DEFAULT ***
Q1 61.00	95.50	30.0	*** METH DEFAULT ***
Q2 98.00	66.90	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	558271
10		10.000	219798
5		5.000	109332
1		1.000	21754
50		50.000	1147128
100		100.000	2455295
0.5	not used for this compound		

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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28) Methyl Acrylate ( )

Ret. Time 10.59 min., Extract & Integrate from 10.09 to 11.09 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 55.00			*** METH DEFAULT ***
Q1 85.00	18.50	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	374149
10		10.000	160341
5		5.000	81772
1		1.000	17877
50		50.000	777455
100		100.000	1646033
0.5	not used for this compound		

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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29) Ethyl tertiary-butyl ether ( )

Ret. Time 10.60 min., Extract & Integrate from 10.10 to 11.10 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 59.00			*** METH DEFAULT ***
Q1 87.00	41.30	30.0	*** METH DEFAULT ***
Q2 57.00	32.40	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	1452475
10		10.000	584977
5		5.000	267663
1		1.000	46902

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50	50.000	3036504
100	100.000	6314530
0.5	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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30) 2,2-Dichloropropane ( )

Ret. Time 10.60 min., Extract & Integrate from 10.10 to 11.10 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 77.00			*** METH DEFAULT ***
Q1 97.00	28.60	30.0	*** METH DEFAULT ***
Q2 41.00	112.10	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
25	25.000	710782
10	10.000	293585
5	5.000	146387
1	1.000	30152
50	50.000	1349364
100	100.000	2709502
0.5	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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31) Bromochloromethane ( )

Ret. Time 10.39 min., Extract & Integrate from 9.89 to 10.89 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 128.00			*** METH DEFAULT ***
Q1 49.00	140.90	30.0	*** METH DEFAULT ***
Q2 130.00	130.70	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
25	25.000	249703
10	10.000	106943
5	5.000	49929
1	1.000	11192
50	50.000	535335
100	100.000	1137973
0.5	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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32) Tetrahydrofuran ( )

Ret. Time 11.00 min., Extract & Integrate from 10.50 to 11.50 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 42.00			*** METH DEFAULT ***
Q1 72.00	28.80	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
25	25.000	88497
10	10.000	39414
5	5.000	22651
1	1.000	-1
50	50.000	174067
100	100.000	441670
0.5	not used for this compound	

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Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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33) Chloroform ( )

Ret. Time 10.48 min., Extract & Integrate from 9.98 to 10.98 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	83.00			*** METH DEFAULT ***
Q1	85.00	67.60	30.0	*** METH DEFAULT ***
Q2	47.00	26.00	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	995164
10		10.000	420641
5		5.000	198177
1		1.000	46237
50		50.000	1982965
100		100.000	4108998
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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34) Dibromofluoromethane(SURR) ( )

Ret. Time 10.66 min., Extract & Integrate from 10.16 to 11.16 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	111.00			*** METH DEFAULT ***
Q1	113.00	98.00	30.0	*** METH DEFAULT ***
Q2	192.00	9.60	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	561295
10		10.000	234152
5		5.000	115870
1		1.000	25783
50		50.000	1136923
100		100.000	2389781
0.5		0.500	13072

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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35) 1-Chlorobutane ( )

Ret. Time 11.63 min., Extract & Integrate from 11.13 to 12.13 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	56.00			*** METH DEFAULT ***
Q1	41.00	71.30	30.0	*** METH DEFAULT ***
Q2	43.00	34.30	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	1098968
10		10.000	440748
5		5.000	202368
1		1.000	39169
50		50.000	2282885
100		100.000	4790439
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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36) 1,1,1-Trichloroethane ( )

Ret. Time 11.64 min., Extract & Integrate from 11.14 to 12.14 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 97.00			*** METH DEFAULT ***
Q1 99.00	66.20	30.0	*** METH DEFAULT ***
Q2 61.00	34.90	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
25	25.000	721155
10	10.000	293809
5	5.000	147853
1	1.000	30957
50	50.000	1440140
100	100.000	2962268
0.5	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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37) 1,1-Dichloropropene ( )

Ret. Time 11.94 min., Extract & Integrate from 11.44 to 12.44 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 75.00			*** METH DEFAULT ***
Q1 110.00	50.40	30.0	*** METH DEFAULT ***
Q2 77.00	31.70	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
25	25.000	632648
10	10.000	249432
5	5.000	115808
1	1.000	23846
50	50.000	1371122
100	100.000	2910338
0.5	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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38) Cyclohexane ( )

Ret. Time 12.07 min., Extract & Integrate from 11.57 to 12.57 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 56.00			*** METH DEFAULT ***
Q1 84.00	79.60	30.0	*** METH DEFAULT ***
Q2 41.00	70.50	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
25	25.000	907077
10	10.000	355664
5	5.000	169423
1	1.000	35456
50	50.000	1863476
100	100.000	3891957
0.5	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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39) Carbon Tetrachloride ( )

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Ret. Time 12.21 min., Extract & Integrate from 12.01 to 12.41 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 117.00			*** METH DEFAULT ***
Q1 119.00	95.40	30.0	*** METH DEFAULT ***
Q2 121.00	32.30	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	535324
10		10.000	216339
5		5.000	101142
1		1.000	21638
50		50.000	1109861
100		100.000	2361524
0.5	not used for this compound		

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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40) Benzene ( )

Ret. Time 12.28 min., Extract & Integrate from 11.78 to 12.78 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 78.00			*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	1904939
10		10.000	784884
5		5.000	378428
1		1.000	70104
50		50.000	3854560
100		100.000	8180736
0.5	not used for this compound		

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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41) 1,2-Dichloroethane-d4(SURR) ( )

Ret. Time 11.38 min., Extract & Integrate from 10.88 to 11.88 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 65.00			*** METH DEFAULT ***
Q1 102.00	38.00	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	589788
10		10.000	252323
5		5.000	131353
1		1.000	31041
50		50.000	1195026
100		100.000	2489303
0.5		0.500	14936

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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42) 1,2-Dichloroethane ( )

Ret. Time 11.49 min., Extract & Integrate from 11.29 to 11.69 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 62.00			*** METH DEFAULT ***
Q1 98.00	15.80	30.0	*** METH DEFAULT ***
Q2 49.00	46.90	30.0	*** METH DEFAULT ***

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Lvl	ID	Conc (ug/l)	Response
25		25.000	728730
10		10.000	312418
5		5.000	158587
1		1.000	36484
50		50.000	1471334
100		100.000	3064482
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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43) Tertiary-amyl methyl ether ( )

Ret. Time 12.55 min., Extract & Integrate from 12.05 to 13.05 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	73.00			*** METH DEFAULT ***
Q1	43.00	42.10	30.0	*** METH DEFAULT ***
Q2	55.00	23.60	30.0	*** METH DEFAULT ***
Q3	87.00	26.10	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	909250
10		10.000	381598
5		5.000	178280
1		1.000	28156
50		50.000	1984661
100		100.000	4429119
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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44) Trichloroethene ( )

Ret. Time 13.29 min., Extract & Integrate from 12.79 to 13.79 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	95.00			*** METH DEFAULT ***
Q1	97.00	66.10	30.0	*** METH DEFAULT ***
Q2	130.00	130.10	30.0	*** METH DEFAULT ***
Q3	132.00	124.60	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	525298
10		10.000	209767
5		5.000	101671
1		1.000	20367
50		50.000	1098688
100		100.000	2293127
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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45) 1,2-Dichloropropane ( )

Ret. Time 13.22 min., Extract & Integrate from 12.72 to 13.72 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	63.00			*** METH DEFAULT ***
Q1	112.00	6.00	30.0	*** METH DEFAULT ***
Q2	61.00	14.20	30.0	*** METH DEFAULT ***

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Lvl	ID	Conc (ug/l)	Response
25		25.000	612692
10		10.000	260437
5		5.000	122156
1		1.000	28518
50		50.000	1253960
100		100.000	2644077
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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46) Dibromomethane ( )

Ret. Time 13.16 min., Extract & Integrate from 12.66 to 13.66 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	93.00			*** METH DEFAULT ***
Q1	95.00	85.70	30.0	*** METH DEFAULT ***
Q2	174.00	84.70	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	299812
10		10.000	121141
5		5.000	62564
1		1.000	14370
50		50.000	632274
100		100.000	1349994
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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47) 2-Nitropropane ( )

Ret. Time 13.31 min., Extract & Integrate from 12.81 to 13.81 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	43.00			*** METH DEFAULT ***
Q1	41.00	89.50	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	115216
10		10.000	46205
5		5.000	26023
1		1.000	4671
50		50.000	238414
100		100.000	420606
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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48) Bromodichloromethane ( )

Ret. Time 13.35 min., Extract & Integrate from 12.85 to 13.85 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	83.00			*** METH DEFAULT ***
Q1	85.00	62.60	30.0	*** METH DEFAULT ***
Q2	127.00	12.70	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	699569
10		10.000	281122
5		5.000	129756

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1	1.000	30183
50	50.000	1384589
100	100.000	2844463
0.5	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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49) 1,4-Dioxane ( )

Ret. Time 13.56 min., Extract & Integrate from 13.06 to 14.06 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 88.00			*** METH DEFAULT ***
Q1 58.00	56.10	30.0	*** METH DEFAULT ***
Q2 43.00	70.10	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		500.000	33325
10		200.000	22779
5		100.000	11540
1		20.000	6822
50		1000.000	74642
100		1500.000	187314
0.5	not used for this compound		

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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50) Methyl Methacrylate ( )

Ret. Time 13.62 min., Extract & Integrate from 13.12 to 14.12 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 41.00			*** METH DEFAULT ***
Q1 69.00	57.10	30.0	*** METH DEFAULT ***
Q2 100.00	31.30	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	426428
10		10.000	161949
5		5.000	74582
1		1.000	-1
50		50.000	976712
100		100.000	2135220
0.5	not used for this compound		

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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51) 2-Chloroethyl vinyl ether ( )

Ret. Time 14.04 min., Extract & Integrate from 13.54 to 14.54 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 63.00			*** METH DEFAULT ***
Q1 65.00	30.70	30.0	*** METH DEFAULT ***
Q2 106.00	34.60	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		125.000	1201319
10		50.000	472654
5		25.000	194776
1		5.000	-1
50		250.000	2677002
100		500.000	6006774

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0.5 not used for this compound

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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52) Methyl Cyclohexane ( )

Ret. Time 14.10 min., Extract & Integrate from 13.60 to 14.60 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	83.00			*** METH DEFAULT ***
Q1	55.00	85.60	30.0	*** METH DEFAULT ***
Q2	98.00	52.50	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	573799
10		10.000	226672
5		5.000	119751
1		1.000	19461
50		50.000	1188169
100		100.000	2513523
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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53) 4-Methyl-2-Pentanone ( )

Ret. Time 14.59 min., Extract & Integrate from 14.39 to 14.79 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	58.00			*** METH DEFAULT ***
Q1	43.00	312.30	30.0	*** METH DEFAULT ***
Q2	85.00	38.90	30.0	*** METH DEFAULT ***
Q3	100.00	43.10	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		125.000	628870
10		50.000	261111
5		25.000	114609
1		5.000	29151
50		250.000	1393379
100		500.000	3030687
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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54) cis-1,3-Dichloropropene ( )

Ret. Time 14.39 min., Extract & Integrate from 13.89 to 14.89 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	75.00			*** METH DEFAULT ***
Q1	77.00	31.40	30.0	*** METH DEFAULT ***
Q2	39.00	53.80	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	677818
10		10.000	257094
5		5.000	120916
1		1.000	23281
50		50.000	1483726
100		100.000	3268559
0.5		0.500	11596

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Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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55) trans-1,3-Dichloropropene ( )

Ret. Time 15.08 min., Extract & Integrate from 14.58 to 15.58 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 75.00			*** METH DEFAULT ***
Q1 77.00	31.90	30.0	*** METH DEFAULT ***
Q2 39.00	61.90	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	553563
10		10.000	216459
5		5.000	91643
1		1.000	22578
50		50.000	1252755
100		100.000	2813004
0.5		0.500	7469

Qualifier Peak Analysis ON
Curve Fit: Linear

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56) 1,1,2-Trichloroethane ( )

Ret. Time 15.33 min., Extract & Integrate from 14.83 to 15.83 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 83.00			*** METH DEFAULT ***
Q1 97.00	126.30	30.0	*** METH DEFAULT ***
Q2 85.00	62.40	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	325064
10		10.000	139694
5		5.000	69384
1		1.000	13659
50		50.000	682634
100		100.000	1473009
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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57) Toluene ( )

Ret. Time 15.66 min., Extract & Integrate from 15.16 to 16.16 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 92.00			*** METH DEFAULT ***
Q1 91.00	160.60	30.0	*** METH DEFAULT ***
Q2 65.00	14.20	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	1086020
10		10.000	439079
5		5.000	197248
1		1.000	35619
50		50.000	2260646
100		100.000	4739947
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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58) Chlorobenzene-d5 (ISTD)

Ret. Time 17.92 min., Extract & Integrate from 17.42 to 18.42 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 117.00			*** METH DEFAULT ***
Q1 119.00	31.70	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
25	25.000	1149114
10	25.000	1152088
5	25.000	1198151
1	25.000	1051571
50	25.000	1180885
100	25.000	1210800
0.5	25.000	1056414

Qualifier Peak Analysis ON ISTD conc: 25.000 ug/l
Curve Fit: Avg. RF

---

59) Toluene-d8 (SURR) ( )

Ret. Time 15.54 min., Extract & Integrate from 15.04 to 16.04 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 98.00			*** METH DEFAULT ***
Q1 100.00	72.40	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
25	25.000	1457463
10	10.000	566890
5	5.000	248627
1	1.000	44328
50	50.000	3183076
100	100.000	6956506
0.5	0.500	20355

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

60) Ethyl Methacrylate ( )

Ret. Time 15.79 min., Extract & Integrate from 15.29 to 16.29 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 69.00			*** METH DEFAULT ***
Q1 41.00	91.80	30.0	*** METH DEFAULT ***
Q2 99.00	27.60	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
25	25.000	317915
10	10.000	120387
5	5.000	54326
1	1.000	-1
50	50.000	729766
100	100.000	1581816
0.5	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

61) 2-Hexanone ( )

Ret. Time 15.99 min., Extract & Integrate from 15.49 to 16.49 min.

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Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	43.00			*** METH DEFAULT ***
Q1	58.00	44.90	30.0	*** METH DEFAULT ***
Q2	57.00	12.90	30.0	*** METH DEFAULT ***
Q3	100.00	9.80	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		125.000	1400232
10		50.000	572507
5		25.000	264854
1		5.000	52715
50		250.000	2993528
100		500.000	6284389
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

62) 1,3-Dichloropropane ( )

Ret. Time 15.73 min., Extract & Integrate from 15.53 to 15.93 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	76.00			*** METH DEFAULT ***
Q1	78.00	33.90	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	636645
10		10.000	276904
5		5.000	139114
1		1.000	25367
50		50.000	1328764
100		100.000	2671607
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

63) Tetrachloroethene ( )

Ret. Time 16.85 min., Extract & Integrate from 16.35 to 17.35 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	164.00			*** METH DEFAULT ***
Q1	129.00	119.50	30.0	*** METH DEFAULT ***
Q2	131.00	111.80	30.0	*** METH DEFAULT ***
Q3	166.00	126.10	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	377976
10		10.000	148543
5		5.000	73358
1		1.000	16445
50		50.000	804360
100		100.000	1781312
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

64) Dibromochloromethane ( )

Ret. Time 16.16 min., Extract & Integrate from 15.66 to 16.66 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	129.00			*** METH DEFAULT ***

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Q1	127.00	75.20	30.0	*** METH DEFAULT ***
Q2	131.00	24.50	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	392387
10		10.000	153553
5		5.000	74019
1		1.000	13781
50		50.000	863711
100		100.000	1921566
0.5		0.500	6279

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

65) 1,2-Dibromoethane ( )

Ret. Time 16.57 min., Extract & Integrate from 16.07 to 17.07 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	107.00			*** METH DEFAULT ***
Q1	109.00	95.30	30.0	*** METH DEFAULT ***
Q2	188.00	1.90	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	353284
10		10.000	146149
5		5.000	70101
1		1.000	12382
50		50.000	796363
100		100.000	1791032
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

66) 1-Chlorohexane ( )

Ret. Time 17.86 min., Extract & Integrate from 17.36 to 18.36 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	91.00			*** METH DEFAULT ***
Q1	93.00	33.30	30.0	*** METH DEFAULT ***
Q2	55.00	55.90	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	495953
10		10.000	171862
5		5.000	76808
1		1.000	15541
50		50.000	1082560
100		100.000	-1
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

67) Chlorobenzene ( )

Ret. Time 17.98 min., Extract & Integrate from 17.48 to 18.48 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	112.00			*** METH DEFAULT ***
Q1	77.00	37.00	30.0	*** METH DEFAULT ***
Q2	114.00	32.80	30.0	*** METH DEFAULT ***

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Lvl	ID	Conc (ug/l)	Response
25		25.000	1174907
10		10.000	465819
5		5.000	228308
1		1.000	50309
50		50.000	2445956
100		100.000	5301175
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

68) 1,1,1,2-Tetrachloroethane ( )

Ret. Time 17.85 min., Extract & Integrate from 17.35 to 18.35 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	131.00			*** METH DEFAULT ***
Q1	133.00	93.40	30.0	*** METH DEFAULT ***
Q2	119.00	63.20	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	391021
10		10.000	158174
5		5.000	79069
1		1.000	16868
50		50.000	846551
100		100.000	1888814
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

69) Ethylbenzene ( )

Ret. Time 18.32 min., Extract & Integrate from 17.82 to 18.82 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	91.00			*** METH DEFAULT ***
Q1	106.00	35.90	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	1893065
10		10.000	726723
5		5.000	311946
1		1.000	52033
50		50.000	4103410
100		100.000	8979616
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Linear

---

70) Xylene P,M ( )

Ret. Time 18.66 min., Extract & Integrate from 18.16 to 19.16 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	106.00			*** METH DEFAULT ***
Q1	91.00	170.30	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		50.000	1422204
10		20.000	550441
5		10.000	240023
1		2.000	35277

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50	100.000	3044004
100	200.000	6571882
0.5	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Linear

---

71) Xylene O ( )

Ret. Time 19.42 min., Extract & Integrate from 18.92 to 19.92 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 106.00			*** METH DEFAULT ***
Q1 91.00	180.90	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	693942
10		10.000	278413
5		5.000	122731
1		1.000	17454
50		50.000	1469054
100		100.000	3137818
0.5	not used for this compound		

Qualifier Peak Analysis ON
Curve Fit: Linear

---

72) Styrene ( )

Ret. Time 19.29 min., Extract & Integrate from 18.79 to 19.79 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 104.00			*** METH DEFAULT ***
Q1 78.00	30.20	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	1099006
10		10.000	397107
5		5.000	160783
1		1.000	24153
50		50.000	2427566
100		100.000	5388566
0.5	not used for this compound		

Qualifier Peak Analysis ON
Curve Fit: Linear

---

73) Bromoform ( )

Ret. Time 18.86 min., Extract & Integrate from 18.36 to 19.36 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 173.00			*** METH DEFAULT ***
Q1 175.00	46.90	30.0	*** METH DEFAULT ***
Q2 254.00	4.00	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	247606
10		10.000	94230
5		5.000	44415
1		1.000	9375
50		50.000	557107
100		100.000	1276653
0.5	not used for this compound		

Qualifier Peak Analysis ON

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Curve Fit: Avg. RF

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74) cis-1,4-Dichloro-2-butene ( )

Ret. Time 19.11 min., Extract & Integrate from 18.61 to 19.61 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	75.00			*** METH DEFAULT ***
Q1	53.00	79.90	30.0	*** METH DEFAULT ***
Q2	88.00	85.10	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	70035
10		10.000	31849
5		5.000	14487
1		1.000	3206
50		50.000	177757
100		100.000	-1
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

75) Bromofluorobenzene (SURR) ( )

Ret. Time 20.20 min., Extract & Integrate from 19.70 to 20.70 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	95.00			*** METH DEFAULT ***
Q1	174.00	68.90	30.0	*** METH DEFAULT ***
Q2	176.00	63.40	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	618813
10		10.000	249771
5		5.000	113774
1		1.000	20232
50		50.000	1303299
100		100.000	2673692
0.5		0.500	8751

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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76) 1,4 Dichlorobenzene-D4 (ISTD)

Ret. Time 22.11 min., Extract & Integrate from 21.61 to 22.61 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	152.00			*** METH DEFAULT ***
Q1	115.00	76.60	30.0	*** METH DEFAULT ***
Q2	150.00	156.60	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	588311
10		25.000	569637
5		25.000	572894
1		25.000	527165
50		25.000	605755
100		25.000	644006
0.5		25.000	505141

Qualifier Peak Analysis ON ISTD conc: 25.000 ug/l
Curve Fit: Avg. RF

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77) Trans-1,4-Dichloro-2-Butene ( )

Ret. Time 19.75 min., Extract & Integrate from 19.25 to 20.25 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 53.00			*** METH DEFAULT ***
Q1 88.00	52.00	30.0	*** METH DEFAULT ***
Q2 75.00	147.90	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	121372
10		10.000	51739
5		5.000	25474
1		1.000	3142
50		50.000	227308
100		100.000	503763
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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78) 1,2,3-Trichloropropane ( )

Ret. Time 19.69 min., Extract & Integrate from 19.19 to 20.19 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 75.00			*** METH DEFAULT ***
Q1 77.00	32.10	30.0	*** METH DEFAULT ***
Q2 110.00	46.70	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	322230
10		10.000	130377
5		5.000	61639
1		1.000	16957
50		50.000	636122
100		100.000	1399251
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Linear

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79) Isopropylbenzene ( )

Ret. Time 20.14 min., Extract & Integrate from 19.64 to 20.64 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 105.00			*** METH DEFAULT ***
Q1 120.00	27.20	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	1594156
10		10.000	590306
5		5.000	243620
1		1.000	33892
50		50.000	3528560
100		100.000	7744816
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Linear

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80) Bromobenzene ( )

Ret. Time 20.55 min., Extract & Integrate from 20.05 to 21.05 min.

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Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	156.00			*** METH DEFAULT ***
Q1	77.00	104.60	30.0	*** METH DEFAULT ***
Q2	158.00	98.30	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	487021
10		10.000	186519
5		5.000	81412
1		1.000	15101
50		50.000	1085416
100		100.000	2405392
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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81) 1,1,2,2-Tetrachloroethane ( )

Ret. Time 19.41 min., Extract & Integrate from 18.91 to 19.91 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	83.00			*** METH DEFAULT ***
Q1	131.00	12.30	30.0	*** METH DEFAULT ***
Q2	85.00	63.10	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	417593
10		10.000	181461
5		5.000	88743
1		1.000	21859
50		50.000	895802
100		100.000	1844826
0.5		0.500	11426

Qualifier Peak Analysis ON
Curve Fit: Linear

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82) n-Propylbenzene ( )

Ret. Time 20.87 min., Extract & Integrate from 20.37 to 21.37 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	91.00			*** METH DEFAULT ***
Q1	120.00	25.90	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	2113209
10		10.000	787297
5		5.000	337422
1		1.000	49059
50		50.000	4566162
100		100.000	9961710
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Linear

---

83) 2-Chlorotoluene ( )

Ret. Time 21.00 min., Extract & Integrate from 20.50 to 21.50 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	91.00			*** METH DEFAULT ***
Q1	126.00	41.80	30.0	*** METH DEFAULT ***

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Lvl	ID	Conc (ug/l)	Response
25		25.000	1353830
10		10.000	539378
5		5.000	239869
1		1.000	40555
50		50.000	2800815
100		100.000	5954898
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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84) 4-Chlorotoluene ( )

Ret. Time 21.12 min., Extract & Integrate from 20.62 to 21.62 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	91.00			*** METH DEFAULT ***
Q1	126.00	42.70	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	1420409
10		10.000	558637
5		5.000	249781
1		1.000	39577
50		50.000	2921318
100		100.000	6131601
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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85) 1,3,5-Trimethylbenzene ( )

Ret. Time 21.31 min., Extract & Integrate from 20.81 to 21.81 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	105.00			*** METH DEFAULT ***
Q1	120.00	58.70	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	1362287
10		10.000	526400
5		5.000	222983
1		1.000	28916
50		50.000	2882721
100		100.000	6254356
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Linear

---

86) Pentachloroethane ( )

Ret. Time 21.39 min., Extract & Integrate from 20.89 to 21.89 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	119.00			*** METH DEFAULT ***
Q1	117.00	105.30	30.0	*** METH DEFAULT ***
Q2	167.00	84.50	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	288430
10		10.000	119191
5		5.000	56051
1		1.000	11799

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50	50.000	616309
100	100.000	1338507
0.5	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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87) tert-Butylbenzene ( )

Ret. Time 21.68 min., Extract & Integrate from 21.18 to 22.18 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 119.00			*** METH DEFAULT ***
Q1 91.00	52.00	30.0	*** METH DEFAULT ***
Q2 134.00	23.50	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	1093230
10		10.000	422799
5		5.000	175015
1		1.000	23912
50		50.000	2385444
100		100.000	5211855
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Linear

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88) 1,2,4-Trimethylbenzene ( )

Ret. Time 21.83 min., Extract & Integrate from 21.33 to 22.33 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 105.00			*** METH DEFAULT ***
Q1 120.00	50.30	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	1427065
10		10.000	544152
5		5.000	238792
1		1.000	32487
50		50.000	3002805
100		100.000	6409908
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Linear

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89) sec-Butylbenzene ( )

Ret. Time 21.98 min., Extract & Integrate from 21.48 to 22.48 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 105.00			*** METH DEFAULT ***
Q1 134.00	20.40	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	1552406
10		10.000	623645
5		5.000	266416
1		1.000	39345
50		50.000	3273552
100		100.000	7095447
0.5		not used for this compound	

Qualifier Peak Analysis ON

Uncontrolled Document

Curve Fit: Avg. RF

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90) 1,3 Dichlorobenzene ( )

Ret. Time 22.07 min., Extract & Integrate from 21.57 to 22.57 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 146.00			*** METH DEFAULT ***
Q1 111.00	42.50	30.0	*** METH DEFAULT ***
Q2 148.00	64.50	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	868503
10		10.000	352801
5		5.000	158563
1		1.000	34723
50		50.000	1824661
100		100.000	3967013
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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91) 4-Isopropyltoluene ( )

Ret. Time 22.22 min., Extract & Integrate from 21.72 to 22.72 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 119.00			*** METH DEFAULT ***
Q1 134.00	22.60	30.0	*** METH DEFAULT ***
Q2 91.00	18.70	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	1378866
10		10.000	529417
5		5.000	238557
1		1.000	38649
50		50.000	2886552
100		100.000	6026376
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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92) 1,4 Dichlorobenzene ( )

Ret. Time 22.16 min., Extract & Integrate from 21.66 to 22.66 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 146.00			*** METH DEFAULT ***
Q1 111.00	41.20	30.0	*** METH DEFAULT ***
Q2 148.00	63.70	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	899117
10		10.000	367083
5		5.000	178481
1		1.000	39315
50		50.000	1815588
100		100.000	4043026
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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93) n-Butylbenzene ( )

Ret. Time 22.77 min., Extract & Integrate from 22.27 to 23.27 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 91.00			*** METH DEFAULT ***
Q1 92.00	59.50	30.0	*** METH DEFAULT ***
Q2 134.00	27.60	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	1220286
10		10.000	481734
5		5.000	216979
1		1.000	36480
50		50.000	2534892
100		100.000	5411888
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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94) 1,2 Dichlorobenzene ( )

Ret. Time 22.64 min., Extract & Integrate from 22.14 to 23.14 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 146.00			*** METH DEFAULT ***
Q1 111.00	42.90	30.0	*** METH DEFAULT ***
Q2 148.00	63.90	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	764113
10		10.000	305758
5		5.000	139625
1		1.000	28586
50		50.000	1623859
100		100.000	3622555
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

95) 1,2-Dibromo-3-Chloropropane ( )

Ret. Time 23.26 min., Extract & Integrate from 22.76 to 23.76 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 75.00			*** METH DEFAULT ***
Q1 155.00	129.90	30.0	*** METH DEFAULT ***
Q2 157.00	166.10	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	59974
10		10.000	26954
5		5.000	14735
1		1.000	-1
50		50.000	122449
100		100.000	268077
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

96) Hexachloroethane ( )

Ret. Time 23.35 min., Extract & Integrate from 22.85 to 23.85 min.

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Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	117.00			*** METH DEFAULT ***
Q1	119.00	98.30	30.0	*** METH DEFAULT ***
Q2	201.00	35.70	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	241403
10		10.000	98031
5		5.000	44965
1		1.000	8703
50		50.000	541780
100		100.000	1239041
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

97) 1,2,4-Trichlorobenzene ( )

Ret. Time 25.18 min., Extract & Integrate from 24.68 to 25.68 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	180.00			*** METH DEFAULT ***
Q1	182.00	88.60	30.0	*** METH DEFAULT ***
Q2	145.00	39.70	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	450909
10		10.000	185804
5		5.000	83302
1		1.000	17048
50		50.000	995540
100		100.000	2279041
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

98) Hexachlorobutadiene ( )

Ret. Time 25.65 min., Extract & Integrate from 25.15 to 26.15 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	225.00			*** METH DEFAULT ***
Q1	223.00	59.00	30.0	*** METH DEFAULT ***
Q2	227.00	59.90	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	174024
10		10.000	78851
5		5.000	39992
1		1.000	10151
50		50.000	366529
100		100.000	831959
0.5		0.500	3888

Qualifier Peak Analysis ON
Curve Fit: Linear

---

99) Naphthalene ( )

Ret. Time 25.57 min., Extract & Integrate from 25.07 to 26.07 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	128.00			*** METH DEFAULT ***

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Lvl	ID	Conc (ug/l)	Response
25		25.000	706440
10		10.000	293145
5		5.000	128247
1		1.000	27241
50		50.000	1634760
100		100.000	3893262
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

100) 1,2,3-Trichlorobenzene ( )

Ret. Time 25.88 min., Extract & Integrate from 25.38 to 26.38 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	180.00			*** METH DEFAULT ***
Q1	182.00	90.30	30.0	*** METH DEFAULT ***
Q2	145.00	41.40	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
25		25.000	369683
10		10.000	161568
5		5.000	77129
1		1.000	16679
50		50.000	828650
100		100.000	1900077
0.5		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

END OF DATA ANALYSIS PARAMETERS

Wed Aug 31 16:40:37 2005

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Attachment C

ESS LABORATORY MS-3 RUN LOG

BATCH DATE	VIAL #	FILE #	LAB ID	METHOD	COMMENTS / DILUTION /STANDARD ID	ANALYST
		M3				
		M3				
		M3				
		M3				
		M3				
		M3				
		M3				
		M3				
		M3				
		M3				
		M3				
		M3				
		M3				
		M3				
		M3				
		M3				
		M3				
		M3				
		M3				

Run Sequence Confirmation_____

Surrogate:_____

Control Number 20.0020-0604A

On-column IS:_____

All Standards must be noted with a primary or secondary ID

Page_____

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VOA Analysis Map

Analysis _____ **ESS Work Order/Samples** _____

ESS Sample ID	Instr	Directory[1]	File #	Dil	Batch	Sequence	Cal #	RPT	Samp wt/ Vol/ Comments

Comments[2]: _____

VOC Spike ID: _____ **Spike Amt:** _____ **Final Volume:** _____

1 Q:/VOA/"Instr"/
2 All QC SOP compliant unless otherwise noted.
Control # 20.0027-0901a

Analyst_____ Date:____

Reviewed by:_____(second review includes a check and approval of manual integrations)

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Table II A-1: Specific QC Requirements and Performance Standards for VOCs (SW-846 8260B) Using WSC-CAM-II A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
Initial Demonstration of Proficiency	Laboratory Analytical Accuracy & Precision	(1) Must be performed prior to using method on samples. (2) Must be performed for each matrix. (3) Must contain all target analytes. (4) Must follow procedure in Section 8.4 of SW-846 8000B.	No	NA	Refer to Section 8.4 of SW-846 8000B and Section 1.1.2 of this protocol.	NA
GC/MS Tunes with BFB	Inter-laboratory Consistency & Comparability	(1) Criteria listed in Table 4 of SW-846 8260B (the same criteria must be used for all analyses). (2) Every 12 hours prior to sample analysis.	No	NA	Perform instrument maintenance as necessary; retune instrument.	Suspend all analyses until tuning non-compliance is rectified.
Initial Calibration	Laboratory Analytical Accuracy	(1) Must be analyzed at least once prior to analyzing samples, when initial calibration verification or continuing calibration does not meet the performance standards, and when major instrument maintenance is performed. (2) Minimum of 5 standards (or 6 if non-linear regression used). (3) Low standard must be ≤RL. (4) %RSD ≤20, r ≥0.99 (linear regression), or r^2 ≥0.99 (non-linear regression) for each target analyte. (5) If %RSD >20, linear or non-linear regression must be used. (6) Minimum RFs as per Table 4 of SW-846 8260C for lowest concentration standard and for average RF. (7) Must contain all target analytes. (8) Calibration must be performed under the same conditions as the samples (e.g., heated purge). (9) If autosampler used to spike surrogates in calibration standards, one-point	No	RF <0.05; affects nondetect results for affected analyte in all samples analyzed under this initial calibration.	(1) Recalibrate if >10% of target analytes exceed %RSD, "r", or "r^2" criteria. (2) If ≤10% of compounds exceed criteria, recalibration is not required as long as %RSD <40, r >0.98, or r^2 >0.98. (3) If recalculated concentrations from the lowest calibration standard are outside of 70-130% recovery range, either: * The RL must be reported as an estimated value[2], or * The RL must be raised to the concentration of the next highest calibration standard that exhibits acceptable recoveries when recalculated using the final calibration curve.	Sample analysis cannot proceed without a valid initial calibration. Report non-conforming compounds (%RSD >20, r <0.99, r^2 <0.99 or minimum RF not met) in laboratory narrative. If non-linear regression (i.e., quadratic equation) is used for calibration, this must be noted in the laboratory narrative along with the compounds affected.

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Table II A-1: Specific QC Requirements and Performance Standards for VOCs (SW-846 8260B) Using WSC-CAM-II A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
		calibration with 5 standards acceptable for surrogates. (10) If linear or non-linear regression used, verify the RL by recalculating concentrations in lowest calibration standard using the final calibration curve; recoveries must be 70-130%. (11) SIM: Laboratory must monitor a minimum of two ions per analyte (the primary ion or quantitation ion and a minimum of one confirmation ion); this is required for all target analytes, surrogates and internal standards.				
Initial Calibration Verification	Laboratory Analytical Accuracy	(1) Immediately after each initial calibration. (2) Concentration level near midpoint of curve. (3) Prepared using standard source different than used for initial calibration. (4) Must contain all target analytes. (5) Percent recoveries must be between 70-130% for target analytes except for "difficult" analytes[(**)] which must exhibit percent recoveries between 40-160%.	No	NA	Locate source of problem; recalibrate if >10% of all analytes are outside of criteria.	If recovery is outside of 70-130% for any analyte, including "difficult" analytes[(**)], report non-conforming compounds in laboratory narrative.
Continuing Calibration	Laboratory Analytical Accuracy	(1) Every 12 hours prior to the analysis of samples. (2) Concentration level near midpoint of curve. (3) Must contain all target analytes. (4) %D must be ≤20 for each target analyte. (5) Minimum RFs as per Table 4 of SW-846 8260C. (6) Area counts of internal standards in continuing calibration must be between 50 – 200% of the area counts in the associated mid-level initial calibration	No	RF <0.05; affects nondetect results for affected analyte in all samples analyzed under this continuing calibration.	(1) Recalibrate if >20% of target analytes exceed %D criteria. (2) If internal standard is outside of criteria, locate source of problem and reanalyze the continuing calibration. (3) If ≤20% of compounds exceed criteria, recalibration is not required as long as %D	Report non-conforming compounds (%D >20 or minimum RF not met) and associated samples in laboratory narrative.

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Table II A-1: Specific QC Requirements and Performance Standards for VOCs (SW-846 8260B) Using WSC-CAM-II A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
		standard.			<40.	
Method Blank	Laboratory Method Sensitivity (contamination evaluation)	(1) Every 20 samples prior to sample analysis and after calibration standards. (2) Matrix and preservative-specific (e.g., water, methanol). (3) Target analytes must be <RL except for common laboratory contaminants (acetone, methylene chloride, and MEK) which must be <5x the RL.	Yes	NA	(1) If concentration of contaminant in sample is <10x concentration in blank, locate source of contamination; correct problem; reanalyze method blank and associated samples. (2) No corrective action required if concentration of contaminant in sample is >10x concentration in blank or if contaminant not detected in sample.	(1) If sample reanalysis is not possible, report non-conformance in laboratory narrative. (2) If contamination of method blanks is suspected or present, the laboratory, using a "B" or some other convention, should qualify the sample results. Blank contamination should also be documented in the laboratory narrative. (3) If re-analysis is performed within holding time and yields acceptable method blank results, the laboratory may report results of the re-analysis only. (4) If re-analysis is performed outside of holding time, the laboratory must report results of both the initial analysis and re-analysis.
Laboratory Control Sample (LCS)	Laboratory Analytical Accuracy	(1) Every 20 samples or for each new tune clock, whichever is more frequent.	Yes	Recovery <10%; affects nondetect	(1) Locate source of problem; reanalyze LCS	(1) If sample reanalysis is not possible, report non-

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Table II A-1: Specific QC Requirements and Performance Standards for VOCs (SW-846 8260B) Using WSC-CAM-II A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
		(2) Concentration level near midpoint of curve. (3) Must contain all target analytes. (4) Matrix and preservative-specific (e.g., water, methanol). (5) Percent recoveries must be between 70-130% for target analytes except for "difficult" analytes(**) which must exhibit percent recoveries between 40-160%. (6) Can also be used as continuing calibration. NOTE: If used as continuing calibration standard, must be evaluated using Performance Standards, Corrective Actions, and Analytical Response Actions listed above for Continuing Calibration.		results for affected analyte in all samples analyzed under the LCS.	and associated samples if >10% of all analytes are outside of criteria. (2) If ≤10% of compounds are outside of the acceptance criteria, reanalysis is not required as long as recoveries are >10%. (3) If >10% of compounds are above the acceptance criteria (>130%), reanalysis is not required if affected compounds were not detected in associated samples.	conformance in laboratory narrative. (2) If recovery is outside of 70-130% for any analyte, including "difficult" analytes(**), report non-conforming compounds in laboratory narrative. (3) If re-analysis is performed within holding time and yields acceptable LCS results, the laboratory may report results of the re-analysis only. (4) If re-analysis is performed outside of holding time, the laboratory must report results of both the initial analysis and re-analysis.
LCS Duplicate	Laboratory Analytical Accuracy & Precision	(1) Every 20 samples or for each new tune clock, whichever is more frequent. (2) Concentration level near midpoint of curve. (3) Must contain all target analytes. (4) Matrix and preservative-specific (e.g., water, methanol). (5) Percent recoveries must be between 70-130% for target analytes except for "difficult" analytes(**) which must exhibit percent recoveries between 40-160%. (6) Recommended to be run immediately after LCS in analytical sequence. (7) RPDs must be ≤20 for waters and solid.	Yes	Recovery <10%; affects nondetect results for affected analyte in all samples analyzed under this LCS.	(1) Locate source of problem; reanalyze LCS and associated samples if >10% of all analytes are outside of the recovery acceptance criteria. (2) If ≤10% of compounds are outside of the recovery acceptance criteria, reanalysis is not required as long as recoveries are >10%. (3) If >10% of compounds are above the recovery acceptance criteria (>130%), reanalysis is not	(1) If sample reanalysis is not possible, report non-conformance in laboratory narrative. (2) If recovery is outside of 70-130% for any analyte, including "difficult" analytes(**) or RPD >20 for any analyte, including "difficult" analytes**, report non-conforming compounds in laboratory narrative. (3) If re-analysis is performed within holding

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Table II A-1: Specific QC Requirements and Performance Standards for VOCs (SW-846 8260B) Using WSC-CAM-II A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
					required if affected compounds were not detected in associated samples.	time and yields acceptable LCS results, the laboratory may report results of the re-analysis only. (4) If re-analysis is performed outside of holding time, the laboratory must report results of both the initial analysis and re-analysis.
MS/MSD	Method Accuracy & Precision in Sample Matrix	(1) Every 20 samples (at discretion of laboratory or at request of data user). (2) Matrix-specific. (3) Concentration level near midpoint of curve. (4) Must contain all target analytes. (5) Percent recoveries between 70 – 130%. (6) RPDs ≤20 for waters and ≤30 for solids.	Yes ONLY when requested by the data user	Recovery <10%; affects nondetect result for affected analyte in unspiked sample only.	Check LCS; if recoveries are acceptable in LCS, narrate non-conformance.	Note exceedances in laboratory narrative.
Surrogates	Method Accuracy in Sample Matrix	(1) Minimum of 3 surrogates, at retention times across GC run. (2) Percent recoveries must be between 70-130% for individual surrogate compounds.	Yes	Recovery <10%; affects all nondetect VOC results in affected sample.	If one or more surrogates are outside of limits, reanalyze sample unless one of the following exceptions applies: (1) Obvious interference present (e.g., UCM). NOTE: If obvious interference is present and surrogate recovery would cause rejection of data (i.e., <10%), reanalyze sample on dilution. (2) Methanol-preserved samples: re-analysis is not	(1) Report recoveries outside of 70-130% in laboratory narrative. (2) If reanalysis yields similar surrogate non-conformances, the laboratory must report results of both analyses. (3) If reanalysis is performed within holding time and yields acceptable surrogate recoveries, the laboratory may report results of the reanalysis only.

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Table II A-1: Specific QC Requirements and Performance Standards for VOCs (SW-846 8260B) Using WSC-CAM-II A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
					required if % moisture >25 and surrogate recovery is >10%. (3) If one or more surrogates exhibit high recoveries and target analytes are not detected in sample, reanalysis is not required.	(4) If reanalysis is performed outside of the holding time and yields acceptable surrogate recoveries, the laboratory must report results of both analyses. (5) If sample is not re-analyzed due to obvious interference, the laboratory must provide the chromatogram in the data report.
Internal Standards	Laboratory Analytical Accuracy and Method Accuracy in Sample Matrix	(1) Minimum of 3 at retention times across GC run. (2) Area counts in samples must be between 50 – 200% of the area counts in the associated continuing calibration standard. (3) Retention times of internal standards must be within ±30 seconds of retention times in associated continuing calibration standard.	No	Recovery <20%; affects all nondetect results quantitated using affected internal standard in associated sample.	If one or more internal standards are outside of limits, reanalyze sample unless obvious interference present (e.g., UCM). NOTE: If obvious interference is present and internal standard area would cause rejection of data (i.e., <20%), reanalyze sample on dilution.	(1) Report nonconformances in laboratory narrative. Include actual recovery of internal standard and provide summary of analytes quantitated using the internal standard. (2) If reanalysis yields similar internal standard non-conformances, the laboratory must report results of both analyses. (3) If reanalysis is performed within holding time and yields acceptable internal standard recoveries, the laboratory may report results of the reanalysis only. (4) If reanalysis is performed outside of the holding time and yields

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Table II A-1: Specific QC Requirements and Performance Standards for VOCs (SW-846 8260B) Using WSC-CAM-II A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
						acceptable internal standard recoveries, the laboratory must report results of both analyses. (5) If sample is not reanalyzed due to obvious interference, the laboratory must provide the chromatogram in the data report.
Quantitation	NA	(1) Quantitation must be based on internal standard calibration. (2) The laboratory must use the average response factor, linear or non-linear regression curve generated from the associated initial calibration for quantitation of each analyte. (3) The internal standard used for quantitation must be the one nearest the retention time of the subject analyte. (4) Results must be reported with 2 or more "significant figures" if ≥RL. If reporting values below the RL, report with 1 or more "significant figures".[3]	NA	NA	NA	NA
Identification	NA	Refer to SW-846 8260B, Section 7.6.	NA	NA	NA	NA
General Reporting Issues	NA	(1) The laboratory must only report values ≥ the sample-specific reporting limit; optionally, values below the sample-specific reporting limit can be reported as estimated, if requested. The laboratory must report results for samples and blanks in a consistent manner. (2) Dilutions: If diluted and undiluted analyses are performed, the laboratory	NA	NA	NA	(1) Qualification of the data is required if reporting values below the sample-specific reporting limit. (2) Complete analytical documentation for diluted and undiluted analyses must be made available for review

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Table II A-1: Specific QC Requirements and Performance Standards for VOCs (SW-846 8260B) Using WSC-CAM-II A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
		should report results for the lowest dilution within the valid calibration range for <u>each</u> analyte. The associated QC (e.g., method blanks, surrogates, etc.) for each analysis must be reported. (3) Refer to Section 3.3, TICs by GC/MS, for guidance. (4) All soil/sediment sample results preserved in methanol must be corrected for the methanol dilution as per Section 3.2.1 of this CAM protocol. (5) Results for soils/sediments must be reported on a dry-weight basis for comparison to MCP regulatory standards. (6) Refer to Appendix II A-1 for chain-of-custody requirements regarding preservation, cooler temperature, and holding times.				during an audit. (3) TICs will be evaluated at the discretion of the data user consistent with the guidelines presented in Appendix II A–3. (4) The performance of dilutions must be documented in the laboratory narrative or on the report form. Unless due to elevated concentrations of target compounds, reasons for dilutions must be explained in the laboratory narrative. (5) If samples are not properly preserved (pH >2 for aqueous samples, solid samples not completely covered with appropriate preservative) or are not received with an acceptable cooler temperature, note the non-conformances in the laboratory narrative. (6) If samples are preserved and/or analyzed outside of the holding time, note the non-conformances in the laboratory narrative.

** Potentially "difficult" analytes include: acetone, methyl ethyl ketone, 4-methyl-2-pentanone, 2-hexanone, dichlorodifluoromethane, bromomethane, chloromethane, and 1,4-dioxane.

[1]As per Appendix IV of MassDEP Policy #WSC-07-350, *MCP Representativeness Evaluations and Data Usability Assessments*, September 2007, if these results are observed, data users should consider nondetect results as unusable and positive results as estimated with a significant low bias.

[2]If the RL is estimated due to unacceptable recovery of the lowest standard, the CAM RL has not been achieved; Question G of the "MassDEP MCP Analytical Protocol Certification Form" must be answered

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Table II A-1: Specific QC Requirements and Performance Standards for VOCs (SW-846 8260B) Using WSC-CAM-II A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action

"NO" and this must be addressed in the laboratory narrative.

[3]Reporting protocol for "significant figures" is a policy decision included for standardization and consistency for reporting of results and is not a definition of "significant" in the scientific or mathematical sense.

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ESS Laboratory
Division of Thielsch Engineering
Cranston, RI

SOP NO. 30_2009

DETERMINATION OF TRACE ELEMENTS BY STABILIZED TEMPERATURE GRAPHITE FURNACE ATOMIC ABSORPTION

(Methods EPA 200.9, 279.2, SM 3113B 20th Ed, MCP CAM III C and SW846 7000 Series)

APPROVED BY:

Operations Manager	Date 7/28/10
QA Officer	Date 7/14/10
Laboratory Director	Date 7/28/10

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DETERMINATION OF TRACE ELEMENTS BY STABILIZED TEMPERATURE GRAPHITE FURNACE ATOMIC ABSORPTION

(Methods EPA 200.9/200 Series, SM 3113B 20th Ed and SW846 7000 Series)

1.0 SCOPE AND APPLICATION

1.1 This method provides procedures for the determination of dissolved and total recoverable elements by graphite furnace atomic absorption (GFAA) in ground water, surface water, drinking water, storm runoff, industrial and domestic wastewater. This method is also applicable to the determination of total recoverable elements in sediment, sludges, and soil. This method is applicable to the following analytes:

Analyte		Chemical Abstract Services Registry Numbers (CASRN)
Antimony	(Sb)	7440-36-0
Arsenic	(As)	7440-38-2
Cadmium	(Cd)	7440-43-9
Chromium	(Cr)	7440-47-3
Copper	(Cu)	7440-50-8
Lead	(Pb)	7439-92-1
Nickel	(Ni)	7440-02-0
Selenium	(Se)	7782-49-2
Silver	(Ag)	7440-22-4
Thallium	(Tl)	7440-31-5

1.2 For reference where this method is approved for use in compliance monitoring programs [e.g., Clean Water Act (NPDES) or Safe Drinking Water Act (SDWA)], consult both appropriate sections of the Code of Federal Regulation (40 CFR Part 136 Table IB for NPDES, and Part 141 and 141.23 for drinking water), and the latest Federal Register announcements.

1.3 Dissolved analytes can be determined in aqueous samples after suitable filtration and acid preservation.

1.4 For the determination of total recoverable analytes in aqueous and solid samples a digestion/extraction is required prior to analysis when the elements are not in solution (e.g., soils, sludges, sediments and aqueous samples that may contain particulate and suspended solids). Aqueous samples containing suspended or particulate material $\geq$ 1% (w/v) should be extracted as a solid type sample.

1.5 Actual method detection limits and linear working ranges will be dependant on the sample matrix, instrumentation, and selected operating conditions.

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1.6 The sensitivity and limited linear dynamic range (LDR) of GFAA often results in the need to dilute a sample prior to analysis. The actual magnitude of the dilution as well as the cleanliness of the labware used to perform the dilution can dramatically influence the quality of the analytical results. Therefore, sample types requiring large dilutions (> 50: 1) should be analyzed by another approved test procedure which has a larger LDR or which is inherently less sensitive than GFAA.

1.7 Users of the method data should state the data-quality objectives prior to analysis. Users of the method must document and have on file the required initial demonstrations performance data described in section 8 prior to using the method for analysis.

2.0 METHOD SUMMARY

2.1 An aliquot of a well mixed, homogeneous aqueous or solid sample is accurately weighed or measured for sample processing. For total recoverable analysis of a solid or an aqueous sample containing undissolved material, analytes are first solubilized by gently refluxing with nitric and hydrochloric acids. After cooling, the sample is made up to volume and mixed prior to analysis.

2.1.1 For 200 Series and SM 3113B, See SOP 30_3005A

2.1.2 For 7000 series metals

2.1.2.1 Aqueous – See SOP 30_3005A

2.1.2.2 Solids – See SOP 30_3050B

2.2 The analytes listed in this method are determined by stabilized temperature platform graphite furnace atomic absorption (STPGFAA). In STPGFAA, the sample and the matrix modifier are first pipetted onto the platform or a device which provides delayed atomization. The furnace chamber is then purged with a continuous flow of a premixed gas (95% argon/5% hydrogen) and the sample is dried at a relatively low temperature (about 120 °C) to avoid spattering. Once dried, the sample is pretreated in a char or ashing step which is designed to minimize the interference effects caused by the concomitant sample matrix. After the char step, the furnace is allowed to cool prior to atomization. The atomization cycle is characterized by rapid heating of the furnace to a temperature where the metal (analyte) is atomized from the pyrolytic graphite surface into a stopped gas flow atmosphere of argon containing 5% hydrogen. (Selenium is determined in an atmosphere of high purity argon.) The resulting atomic cloud absorbs the element specific atomic emission produced by a hollow cathode lamp (HCL) or an electrodeless discharge lamp (EDL). Following analysis, the furnace is subjected to a cleanout period of high temperature and continuous argon flow. Because the resulting absorbance usually has a nonspecific component associated with the actual analyte absorbance, an instrumental background correction device is used to

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subtract from the total signal the component that is nonspecific to the analyte. Interferences relating to STPGFAA must be recognized and corrected. Suppressions or enhancements of instrument response caused by the sample matrix must be corrected by the method of standard addition.

3.0 HEALTH AND SAFETY

3.1 Each employee has been trained and has acknowledged being trained in the safe use and handling of chemicals being used in the laboratory. This training has been performed according to the ESS Training SOP 80_0016 and by the Chemical Hygiene Plan, SOP No. 90_0001, in conjunction with the Safety orientation.

3.2 All sample and material handling should be done in a hood while using proper protective equipment to minimize exposure to liquid or vapor. Minimum personnel protective equipment includes the use of laboratory safety glasses, a lab coat or apron, and protective gloves.

3.3 The MSDSs for the concentrated chemical used in the area are kept on file in a central location that is available for the employees for review.

3.4 Several chemicals used in this procedure are tentatively classified as known or suspected carcinogens. Extreme caution should be used in handling all chemicals in this procedure.

3.5 Proper emergency response to spills or injury should be reviewed by the laboratory employee prior to attempting this procedure. Employees must know the location of spill kits, eyewashes, showers, and fire fighting equipment. Employees must also have knowledge of disaster evacuation routes.

3.6 The graphite tube during atomization emits intense UV radiation. Suitable precautions should be taken to protect personnel from such a hazard.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

4.1 Prior to collection of an aqueous sample, consideration should be given to the type of data required, (i.e., dissolved or total recoverable), so that appropriate preservation and pretreatment steps can be taken. The pH of all aqueous samples must be tested immediately prior to aliquoting for processing to ensure the sample has been properly preserved. If properly acid preserved, the sample can be held up to 180 days before analysis.

4.2 For the determination of the dissolved elements, the sample is filtered through a 0.45 µm pore diameter membrane filter within 24 hours of collection. Use a portion of the filtered sample to rinse the filter flask, discard this portion and collect the required volume of filtrate. Acidify the filtrate with (1:1) nitric acid immediately following filtration to pH < 2.

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4.3 For the determination of total recoverable elements in aqueous samples, samples are not filtered, but acidified with (1:1) nitric acid to pH <2 (3 ml of 1:1 HNO_3 per liter of sample is sufficient for most ambient and drinking water samples). Preservation is to be performed at the time of collection. Following acidification, the sample should be mixed, held for sixteen hours, and then verified to be pH <2 just prior to withdrawing an aliquot for processing or direct analysis. If for some reason such as high alkalinity the sample pH is verified to be > 2, more acid must be added and the sample held for sixteen hours until verified to be pH < 2.

NOTE: When the nature of the sample is either unknown or is known to be hazardous, acidification should be done in a fume hood.

4.4 Solid samples require no preservation prior to analysis other than storage at 4°C. Solid samples may be held for 180 days.

4.5 For aqueous samples, a field blank should be prepared and analyzed as required by the data user. Use the same type of container and acid as used in sample collection.

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

5.1 Several interference sources may cause inaccuracies in the determination of trace elements by GFAA. These interferences can be classified into three major subdivisions, namely, spectral, matrix, and memory.

5.1.1 Spectral interferences are caused by the resulting absorbance of light by a molecule or atom which is not the analyte of interest or emission from a black body radiation.

5.1.1.1 Spectral interferences caused by an element only occur if there is a spectral overlap between the wavelength of the interfering element and the analyte of interest. Fortunately, this type of interference is relatively uncommon in STPGFAA because of the narrow atomic line widths associated with STPGFAA. In addition, the use of appropriate furnace temperature programs and high spectral purity lamps as light sources can minimize the possibility of this type of interference. However, molecular absorbance can span several hundred nanometers producing broadband spectral interferences. This type of interference is far more common in STPGFAA. The background correctors are all attempts to eliminate unwanted nonspecific absorbance. The nonspecific component of the total absorbance can vary considerably from matrix to matrix. Therefore, the effectiveness of a particular background correction device may vary depending on the actual analyte wavelength used as well as the nature and magnitude of the interference. The background correction device to be used with this method is not specified,

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however, it must provide an analytical condition that is not subject to the occurring interelement spectral interferences of palladium on copper, iron on selenium, and aluminum on arsenic.

5.1.1.2 Spectral interferences are also caused by the emissions from black body radiation produced during the atomization furnace cycle. This black body emission reaches the photomultiplier tube, producing erroneous results. The magnitude of this interference can be minimized by proper furnace tube alignment and monochromator design. In addition, atomization temperatures which adequately volatize the analyte of interest without producing unnecessary black body radiation can help reduce unwanted background emission during analysis.

5.1.2 Matrix interferences are caused by sample components which inhibit the formation of free atomic analyte atoms during the atomization cycle.

5.1.2.1 Matrix interferences can be of a chemical or physical nature. In this method, the use of a delayed atomization device which provides stabilized temperatures is required. These devices provide an environment which is more conducive to the formation of free analyte atoms and thereby minimize this type of interference. This type of interference can be detected by analyzing the sample plus a sample aliquot fortified with a known concentration of the analyte. If the determined concentration of the analyte addition is outside a designated range, a possible matrix effect should be suspected.

5.1.2.2 The use of nitric acid is preferred for GFAA analyses in order to minimize vapor state anionic chemical interferences, however, in this method hydrochloric acid is required to maintain stability in solutions containing silver. When hydrochloric acid is used, the chloride ion vapor state interferences must be reduced using an appropriate matrix modifier. In this method, a combination modifier of palladium, magnesium nitrate and hydrogen (5%) -argon (95%) gas mixture is used for this purpose.

5.1.3 Memory interferences result from analyzing a sample containing a high concentration of an element (typically a high atomization temperature element) which can not be removed quantitatively in one complete set of furnace steps. The analyte which remains in the furnace can produce false positive signals on subsequent sample(s). Therefore, the analyst should establish the analyte concentration which can be injected into the furnace and adequately removed in one complete set of furnace cycles. If this concentration is exceeded, the sample should be diluted and a blank analyzed to assure the memory effect has been eliminated before reanalyzing the diluted sample.

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5.2 Specific element interferences are noted below when a combination modifier of palladium, magnesium nitrate and hydrogen (5%) - argon (95%) gas are used.

5.2.1 Arsenic: The HCl level present from the digestion procedure can influence the sensitivity for As. 20 µL of a 1% HCl solution with Pd used as modifier results in a 20% loss in sensitivity relative to the analyte in a 1% HNO_3 solution. Unfortunately, the use of $Pd/Mg/H_2$ as a modifier does not significantly reduce this suppression, and therefore, it is imperative that each sample and calibration standard alike contain the same HCl concentration.

5.2.2 Lead: The HCl present from the digestion procedure can influence the sensitivity for Pb. 20µL of a 1% HCl solution with Pd used as a modifier results in a 75% loss in sensitivity relative to the analyte response in a 1% HNO_3 solution. The use of $Pd/Mg/H_2$ as a matrix modifier reduces this suppression to less than 10%.

5.2.3 Selenium: Iron has been shown to suppress Se response with continuum background correction. In addition, the use of hydrogen as a purge gas during the dry and char steps can cause suppression in the Se response if not purged from the furnace prior to atomization.

5.2.4 Silver: The palladium used in the modifier preparation may have elevated levels of Ag which will cause elevated blank absorbances.

5.2.5 Thallium: The HCl present from the digestion procedure can influence the sensitivity for Tl. 20ul of a 1% HCl solution with Pd used as a modifier results in a 90% loss in sensitivity relative to the analyte in a 1% HNO_3 solution. The use of $Pd/Mg/H_2$ as a matrix modifier reduces this suppression to less than 10%.

6.0 EQUIPMENT/APPARATUS

6.1 **Graphite Furnace Atomic Absorption Spectrophotometer**:

6.1.1 The GFAA spectrometer must be capable of programmed heating of the graphite tube and the associated delayed atomization device. The instrument must be equipped with an adequate background removing specific correction device capable of removing undesirable non-specific absorbance over the spectral region of interest and provide an analytical condition not subject to the occurrence of interelement spectral overlap interferences. The furnace device must be capable of utilizing an alternate gas supply during specified cycles of the analysis. The capability to record relatively fast (≤1s) transient signals and evaluate data on a peak area basis is preferred. In

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addition, a recirculating refrigeration bath is recommended for improved reproducibility of furnace temperatures.

6.1.2 **Single element hollow cathode lamps** or single element electrodeless discharge lamps along with the associated power supplies.

6.1.3 **Argon gas supply** (high purity grade, 99.99%) for use during the atomization of selenium, for sheathing the furnace tube when in operation, and during furnace cleanout.

6.1.4 **Alternate gas mixture** (hydrogen 5%/argon 95%) for use as a continuous gas flow environment during the dry and char furnace cycles.

6.1.5 **Auto-sampler** capable of adding matrix modifier solutions to the furnace, a single addition of analyte, and completing methods of standard additions when required.

6.2 **Analytical balance**, with capability to measure to 0.1 mg, for use in weighing solids, for preparing standards, and for determining dissolved solids in digests or extracts.

6.3 **Variable transfer pipettes**, 0.025 - 0.1 ml and 0.1 - 1.0 ml calibrated according to SOP 110.0005.

6.4 **Plastic/Glassware**: Volumetric flasks, funnels and centrifuge tubes (glass and/or metal-free plastic), and specimen cups.

6.5 **Volumetric flasks**: Class A in various sizes.

6.6 **Data system**:

6.6.1 **Computers:** The Metals laboratory has a DELL OptiPlex 360 computer with a Windows XP Pro/SP3 operating system for analyzing samples by GFAAS All computer systems are networked to a Windows 2000 server, which is the destination of all files. A differential back-up is performed nightly and a full back is performed each weekend using Veritas Backup Exec to tapes. As the systems acquires and stores data onto the server, the server becomes full. The data is downloaded and archived onto CDs.

6.6.2 **Software:**WINLAB3260 AA Version 6.4

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7.0 REAGENTS AND STANDARDS

7.1 **Reagents**: Reagent grade or better chemicals shall be used in all tests. If the purity of a reagent is in question; it is analyzed for contamination. If the target analytes concentration is less than the MRL, then the reagent is acceptable.

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7.1.1 **Hydrochloric acid** (conc), HCl: Trace Grade

7.1.2 **Hydrochloric acid** (1:1), HCl: Add 500 ml concentrated HCl to 400 ml water and dilute to 1 liter in an appropriate beaker. ***Never add water to acid***.

7.1.3 **Nitric acid** (conc), HNO_3 : Trace Grade

7.1.4 **Hydrogen Peroxide**, 30%

1.1.1 **De-Ionized Water** (DI): The deionized water is prepared using the equipment described in SOP 110.0003 The conductivity must also be checked and recorded according to SOP 110.0003. The lab supervisor needs to be notified if the conductivity is > 1 µmhos/cm. At this point, the supervisor will arrange for the cartridges to be replaced, if deemed necessary.

7.1.5 **ICP solution**: All dilutions (primary standards or samples) are performed using ICP solution. It is prepared by adding 20 ml of nitric acid and 30ml of hydrochloric acid to a liter of reagent water. This solution is also used as the calibration blank.

7.1.6 **Palladium Solution** (1%); commercially prepared by VHG Labs.

7.1.7 **Magnesium Nitrate Solution** (1%); commercially prepared by VHG Labs.

7.1.8 **Ammonium Phosphate Solution** (10%); commercially prepared by VHG Labs.

7.2 **Standards**: All standards are prepared in ICP solution for matrix matching with samples. ICP 3 standard is prepared daily and is used to prepare graphite furnace standards.

7.2.1 **Primary Stock Solutions:** They are stored at room temperature and not used beyond the manufacturer's expiration date.

Standard	Vendor	Elements
Mixed Standard 1	VHG	Ag, Al, As, B, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Mo, Na, Ni, Pb, Sb, Se, Sn, Ti, Tl, V, Zn
ICCV	CPI	Ag, Al, As, B, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Mo, Na, Ni, Pb, Sb, Se, Sn, Ti, Tl, V, Zn

7.2.2 **Working standard solutions** are used to calibrate the instruments. They are prepared daily. Initial analyte concentrations are obtained from the bottle label or the vendor's certificate of analysis.

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7.2.2.1 **ICP Standard 3**: Add 1.0 ml of Mixed Standard 1 (see 7.2.1) to a 100 ml volumetric flask and dilute to the 100 ml mark with ICP solution. Concentration is 1000 ppb except for Ag/Cd (500 ppb), Se (2000 ppb), and Be (100 ppb).

7.2.2.2 The **Instrument Check Standard** (SCV1: initial calibration verification) is used to verify the primary standard. It is prepared by adding 0.5 ml of ICCVA to a 100 ml class A volumetric flask and diluting to the mark with ICP solution.

7.2.2.3 **Mixed standards**: Place a clean 15 ml centrifuge tube into a specimen cup and tare on the balance. Prepare each standard as follows:

7.2.2.3.1 **5 ppb**: Weigh 9.95 grams of ICP solution. Pipette 0.05 ml of ICP 3 Standard (1ppm-see 7.2.2.1) into the tube. Weight should equal 10.00g ± 0.01g.

7.2.2.3.2 **10 ppb**: Weigh 9.90 grams of ICP solution. Pipette 0.10 ml of ICP 3 Standard (1ppm-see 7.2.2.1) into the tube. Weight should equal 10.00g ± 0.01g.

7.2.2.3.3 **25 ppb**: Weigh 9.75 grams of ICP solution. Pipette 0.25 ml of ICP 3 Standard (1ppm-see 7.2.2.1) into the tube. Weight should equal 10.00g ± 0.01g. This standard will also be used as the continuing calibration verification (CCV) standard for the run. It is run immediately after the calibration curve to verify the calibration.

7.2.2.3.4 **50 ppb**: Weigh 9.50 grams of ICP solution. Pipette 0.50 ml of ICP 3 Standard (1ppm-see 7.2.2.2) into the tube. Weight should equal 10.00g ± 0.01g.

7.2.2.3.5 **100 ppb second source standard (used for pds)**: Weigh 8.00 grams of ICP solution. Pipette 2.00 ml of ICV standard into the tube. Weight should equal 10.00g ± 0.01g.

7.2.2.3.6 **25 ppb** SCV1: Weigh 9.50 grams of ICP solution. Pipette 0.50 ml of ICV (0.5ppm-see 7.2.2.2) into the tube. Weight should equal 10.00g ± 0.01g.

7.2.2.3.7 **CRA 2ppb standard**: Pipette 0.4 ml of 50 ppb standard into ICP solution with final volume equal to 10ml.

7.2.2.3.8 **Ag/Cd PDS solution**: A 10 ppb standard is used (5x dilution of Section 7.2.2.3.5 standard.

7.2.2.4 **Silver standards-** Due to the increased sensitivity of silver, the standard curve is prepared at concentrations of 0.5, 1.0, 2.5, and 5.0 ppb. The 5.0ppb is equal to the 10 ppb (7.2.2.3.2), the 2.5 ppb is equal to the 5 ppb (7.2.2.3.1), and the 1ppb standard is equal to the

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CRA standard (7.2.2.3.7). Prepare a two times dilution, using ICP solution to prepare the 0.5ppb CRA standard.

7.2.2.5 **Cadmium standards-** Due to the increased sensitivity of cadmium, the standard curve is prepared at concentrations of 0.1, 0.2, 0.5, and 1.0 ppb. The 1ppb standard is equal to the CRA standard (7.2.2.3.7). Prepare a two times dilution, using ICP solution to prepare the 0.5ppb standard. Prepare a five times dilution, using ICP solution to prepare the 0.2ppb standard. Prepare a ten times dilution, using ICP solution to prepare the 0.1ppb standard.

7.2.3 **Palladium/Magnesium Modifier** - Add 50 ml DI water to a 100 ml volumetric flask. Add 6 ml of 1% Palladium (see 7.1.7) and 10 ml 1% Magnesium Nitrate (see 7.1.8). Volumize to 100 ml and mix.

7.2.4 **Phospate/Magnesium modifier** - Add 50 ml DI water to a 100 ml Flask. Add 6 ml of 1% Magnesium Nitrate (see 7.1.8) and 10 ml of ammonium phosphate (7.1.9). Volumize to 100 ml and mix. This modifier is used for the analysis of lead only instead of the Pd/Mg.

7.2.5 **Blank Spike/Blank Spike Duplicate (BS/BSD)**: To 50 ml of reagent water added to a beaker, add 2 ml of ICV standard (Section 7.2.2.1). This undergoes the same preparation procedure as a typical sample. Final concentration is 20 ppb for most of the analytes listed in section 1.1.

7.2.6 The **matrix spike** is a sample that is measured out, spiked with 2 ml of ICV standard (Section 7.2.2.2), and undergoes the same preparation as the original sample.

7.2.7 The **matrix duplicate** sample is a second aliquot of sample that undergoes the same preparation as the original sample

NOTE: Due to the high sensitivity of Silver and Cadmium, the LCS and MS must be diluted 5x for Silver and 20x for Cadmium in order to get the concentration within the calibration curve.

8.0 PROCEDURE

8.1 **Sample Preparation:** Preliminary treatment of most matrices is necessary because of the complexity and variability of sample matrices.

8.1.1 Water samples which have been prefiltered and acidified will not need acid digestion as long as the samples and standards are matrix matched.

8.1.2 ESS Laboratory policy is to digest dissolved elements samples as any other aqueous metal sample, per SOP 30_3005B.

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8.1.3 For the determination of total recoverable analytes in aqueous samples per 200.9 (other than drinking water with < 1 NTU turbidity), prepare samples according to SOP 30_3005.

8.2 Sample Analysis:

8.2.1 Prior to daily calibration of the instrument, inspect the graphite furnace, the sample uptake system and autosampler injector for any change in the system that would affect instrument performance. Clean the system and replace the graphite tube and/or platform when needed or on a daily basis. Record any maintenance performed in the maintenance log.

8.2.2 Turn on in the following order:

8.2.2.1 Argon gas supply.

8.2.2.2 Argon/Hydrogen gas supply.

8.2.2.3 Spectrometer.

8.2.2.4 Computer and Monitor.

8.2.2.5 Printer.

8.2.3 Select **AAwinlab** analyst icon.

8.2.3.1 Select **Lamp** icon in the toolbar. Select the desired lamp. If lamp is not already loaded, select a turret, remove lamp that is present, and replace with needed lamp. System will set current. If lamp is an EDL, turn on EDL power supply and connect lamp. Set current to recommended level and place lamp in turret.

8.2.3.2 Alignment is achieved when energy line has reached the largest value. Click on the set **Mid-scale** icon. Close **Align Lamps** window.

8.2.4 Select **SampInfo** icon in toolbar. This opens a table for storing the sequence of samples for the autosampler. The following positions are used to hold various standards for the instruments:

Position	GFAA2
Standards	121 - 139
Blank	141, 148
Pd/Mg modifier	145, 147
PO_4/Mg modifier	146

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All other cups may be used for samples. Record all samples in the run log (Attachment B) and then type into Sample ID column. Under analyst, type in your initials. Save table in the format 092798YA for GFAA2, where the first six numbers is the date, the Y stands for the furnace and A is the first analytical run of the day. Close Window.

8.2.5 Select **Method Editor** icon in toolbar. Select the desired method. Select "**Checks**" tab. Select **Automatic Recovery 2** and under **Frequency**, select **All Samples** or **Samples in AS Locations**. Enter in desired Auto-sampler locations for the samples that you want the instrument to spike. **Save Method**. NOTE: *If you are running multiple methods and do not save changes, your entries will not be saved.*

8.2.6 Select **Furnace** icon. If tube needs to be replaced, select open/close, turn screw until sampler swings free, move lower arm out from furnace, pull furnace open, and remove tube with stick of a swab. Clean furnace with ICP Solution, replace new tube, close furnace, replace lower arm, select open/close, swing sampler closed, and turn in screw until finger tight. Record all maintenance in the maintenance log.

8.2.7 Before beginning daily calibration, the instrument system should be reconfigured to the selected optimized operating conditions. Initiate data system and allow a period of not less than 15 minutes for instrument and hollow cathode lamp warm up. If an EDL is to be used, allow 30 minutes for warm up.

8.2.8 After major maintenance (i.e.: Tube replaced, contact rings replaced, etc.) a sensitivity check is performed before initiating calibration. This is also required before running drinking water samples for any method Choose the sensitivity method for the element to be analyzed.

8.2.8.1 Each sensitivity method has been set to analyze 5 replicates of standard 3. The %RSD for the standard must be less that 5% for HCL analytes (Pb, Cu, Tl, Ni, and Ag) and the average sensitivity must be within 20% of the expected value (ug/L). Sensitivity values can be obtained from Perkin Elmer recommended condition for THGA Furnace chart kept in the metals laboratory. Results are kept on file.

8.2.9 For initial and daily operation, calibrate the instrument according to the instrument manufacturer's recommended procedures using the calibration blank and calibration standards prepared at three or more concentrations within the usable linear range of the analyte.

8.2.10 An auto-sampler must be used to introduce all solutions into the graphite furnace. Make sure that there is plenty of DI water in the autosampler rinse

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bottle before starting the run. Also make sure that the waste bottle is emptied. Once the standard, sample or QC solution plus the matrix modifier is injected, the furnace controller completes furnace cycles and cleanout period as programmed. Analyte signals must be integrated and collected as peak area measurements. Background absorbance, background corrected analyte signals, and determined analyte concentrations on all solutions must be able to be displayed on a CRT for immediate review by the analyst and be available electronically for documentation to be kept on file. Flush the autosampler solution uptake system with rinse blank between each solution injected.

8.2.10.1 Using the run log, load appropriate standards and samples into the autosampler tray. In **Automated Analysis** window, open the appropriate SIF. Open results data set: C:\Data – AA\GFAA\ Results\Results.mdb. Name file 010100YAD (GFAA2) and click OK.

8.2.10.2 Select **Analyze** tab. Select **Analyze All**. Instrument will begin calibration process. NOTE: If multiple methods are to be used, you can set delay time between methods to allow for warm-up, the usual delay time between methods should be set to 20-30 minutes. NOTE: GFAA2 is able to warm up 4 lamps at once, which, if used, would mean the delay is unnecessary. Ensure appropriate methods are edited to reflect desired post digestion spikes. Do not forget to save method when making changes to post spike locations. Analysis can be set on desired locations or the **Use Entire SIF** box can be selected. If running analysis overnight or if analyst is not present, select the **Lamp box** to shut lamp off after analysis is completed.

8.2.11 After completion of initial requirements of this method, samples should be analyzed in the same operational manner used in the calibration routine.

8.2.11.1 If calibration is successful, the system will continue with sample analysis. If not, the system will repeat calibration once. If the analyst needs to stop an analytical run, select **Analyze All**. Select **Complete current replicate only** (if sample is in the furnace) or **Stop immediately** (if sampler is in between samples). Then select **OK**.

8.2.12 During the analysis of samples, the laboratory must comply with the required quality control. Only for the determination of dissolved analytes or the "direct analysis" of drinking water with turbidity of ≤1 NTU is the sample digestion step of the Method Blank, Blank Spike/Blank Spike Duplicate, Duplicate and Matrix Spike not required.

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8.2.13 For every new or unusual matrix, when practical, it is highly recommended that an inductively coupled plasma emission spectrometer be used to screen for high element concentration. Information gathered from this may be used to prevent potential damage to the instrument and to better estimate which elements may require analysis by graphite furnace.

8.2.14 Determined sample analyte concentrations that are 90% or more of the upper limit of calibration must be either diluted with acidified reagent water and reanalyzed with concern for memory effects, or determined by another approved test procedure that is less sensitive. Samples with a background absorbance >1 must be appropriately diluted with acidified reagent water and reanalyzed. If the method of standards addition is required, see section 8.3.

8.2.15 When it is necessary to assess operative matrix interference, the test described in section 10.5 is recommended.

8.3 Standard Addition: If the method of standard addition is required, the following procedure is recommended.

8.3.1 The standard addition technique involves preparing new standards in the sample matrix by adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample constituent that enhances or depresses the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interference, which causes a baseline shift. The simplest version of this technique is the single-addition method. The procedure is as follows: Two identical aliquots of the sample solution, each of volume V_x, are taken. To the first (labeled A) is added a small volume V_s, of a standard analyte solution of concentration C_s. To the second (labeled B) is added the same volume V_s of the solvent. The analytical signals of A and B are measured and corrected for non-analyte signals. The unknown sample concentration C_x is calculated:

$$C_x = \frac{S_B V_s C_s}{(S_A - S_B)V_x}$$

Where, S_A and S_B are the analytical signals (corrected for the blank) of solutions A and B respectively. V_s and C_s should be chosen so that S_A is roughly twice S_B on the average. It is best if V_s is made much less than V_x, and thus C_s is much greater than C_x to avoid excess dilution of the sample matrix. If a separation or concentration step is used, the additions are best made first and carried through the entire procedure. For the results from this technique to be valid, the following limitations must be taken into consideration:

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8.3.1.1 The analytical curve must be linear.

8.3.1.2 The chemical form of the analyte added must respond in the same manner as the analyte in the sample.

8.3.1.3 The interference effect must be constant over the working range of concern.

8.3.1.4 The signal must be corrected for any additive interference.

9.0 CALCULATIONS

9.1 For aqueous samples the instrument printout is in µg/L. Divide by 1000, to convert to mg/l.

9.1.1 $$\text{Final conc} = \frac{\text{mg/l (raw)} \times \text{dilution factor} \times \text{final volume}}{\text{Initial weight / volume}}$$

9.1.2 For solids, the value must be adjusted for % solids.

10.0 QUALITY ASSURANCE/QUALITY CONTROL

10.1 All quality control data should be maintained and available for easy reference or inspection.

10.2 The correlation coefficient for the linear curve fit must be $r \geq 0.995$.

10.3 Dilute and reanalyze samples that are more concentrated than the highest calibration standard. The instrument is programmed to automatically dilute and re-analyze samples at a 3x dilution.

10.4 Employ a minimum of one method blank per sample batch to determine if contamination is occurring during sample preparation. The method blank must contain all the reagents and in the same volumes as used in the processing of the samples. It must be carried through the complete procedure as the sample solution used for analysis.

10.4.1 The value for the blank should not exceed the MRL. If the method blank is above the MRL, see section 11.1.3. Refer to Attachments C and D respectively, for DOD QSM and MCP CAM requirements.

10.5 At least one BS/BSD must be analyzed for each batch of samples. The BS/BSD should be prepared and analyzed in the same manner as the samples.

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10.5.1 The BS/BSD recovery range must be within the range of 80-120% (85-115% per 200.9). See section 11.0 for corrective action. Refer to

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Attachments C and D respectively, for DOD-QSM and MCP-CAM requirements.

10.6 It is recommended that whenever a new or unusual sample matrix is encountered, a series of tests be performed prior to reporting concentration data for analyte elements. These tests, as outlined in Steps 10.6.1 and 10.6.2 will ensure the analyst that neither positive nor negative interferences are operating on any of the analyte elements to distort the accuracy of the reported values. ESS Laboratory policy is to perform a serial dilution and post digestion spike (PDS) once per analytical batch.

10.6.1 Post digestion spike addition (DOD: "Recovery Test"; refer to Attachment C), (MCP: not required per Attachment D): An analyte spike added to a portion of a prepared sample, or its dilution, should be recovered to within 85% to 115% of the known value. The spike addition should produce a minimum level of 10 times and a maximum of 100 times the instrumental detection limit. If the spike is not recovered within the specified limits, a matrix effect should be suspected. If the spike is <20% of the sample concentration then serial dilution is used to assess matrix effect.

10.6.2 Serial Dilutions (SD): A 5X (1:4) serial dilution test is performed for an analyte to evaluate matrix interference if the analyte concentration in the undiluted sample is at least 50 times the MDL. Matrix effects are suspected if the RPD of the undiluted and diluted sample is >10%. Refer to Attachments C and D respectively, for DOD-QSM and MCP-CAM requirements.

10.6.3 Post digestion spike and serial dilution are performed on the unspiked sample that was chosen for the duplicate and matrix spike determination.

10.7 Check the instrument standardization by analyzing appropriate check standards as follows:

10.7.1 Verify primary standard with a second source standard (SCV1) after the completion of the calibration run.

10.7.1.1 The results of the SCV1 should agree within 10% of the expected value. If this is outside of the control limits, recalibrate. Refer to Attachment D for MCP-CAM requirements.

10.7.2 Verify calibration, immediately after curve, every 10 samples and at the end of the analytical run, using a calibration blank (See 7.1.6) and a check standard (CCV-identified in the LIMS as ICV1) (See 7.2.2.3.3). Refer to Attachments C and D for DOD-QSM and MCP-CAM requirements.

10.7.2.1 The results of the CCV should agree within 10% of the expected value. Samples run between CCVs which are out of control must

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be re-analyzed. (The initial CCV for method 200.9 must be within 5%; 7000 series methods allow 20% for CCV).

10.7.3 Replicate samples are to be analyzed at a frequency of 10% or per analytical batch, whichever is more frequent. A replicate sample is a sample brought through the whole sample preparation and analytical process in duplicate. The replicate sample is designated by adding the suffix "dup" to the sample number. Refer to Attachments C and D respectively, for DOD-QSM and MCP-CAM requirements.

10.7.3.1 The relative percent difference between replicate determinations is to be calculated as follows:

$$RPD = \frac{D_1 - D_2 \times 100}{(D_1 + D_2)/2}$$

Where:

RPD = relative percent difference
D_1 = first sample value
D_2 = second sample value (replicate)

A control limit of $\pm$20% RPD for aqueous samples and $\pm$35 % for soil/sediment samples shall be used for sample values greater than 5 times the method reporting limit. Refer to Attachments C and D respectively, for DOD-QSM and MCP-CAM requirements.

10.7.4 The matrix spikes are to be analyzed at a frequency of 10% or per analytical batch, whichever is more frequent. Matrix spike recovery is to be within $\pm$25% of the actual value. The spiked sample recovery is calculated as follows:

$$\% \text{ Recovery} = \frac{SSR - SR}{SA} \times 100$$

Where:

SSR = Spiked Sample Result
SR = Sample Result
SA = Spike Added to Sample

A control limit of 25% shall be used. Refer to Attachments C and D respectively, for DOD-QSM and MCP-CAM requirements.

10.8 Initial and Periodic Method QC Demonstrations: The procedures specified in Section 10.7.1 through 10.7.2 must be conducted as an initial demonstration of laboratory capability, prior to the analysis of any samples. Subsequent to this initial demonstration, additional evaluations of this nature should be conducted on a

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periodic basis, in response to changes in instrumentation or operations, and/or in response to confirmed or suspected systems, method, or operational problems.

10.8.1 Accuracy and Precision: To demonstrate initial laboratory capability, analyze a minimum of four initial calibration verification standards. The mean concentration must be within ± 10 % of the stated values.

10.8.2 Method Detection Limits for Individual Analytes: Analyze a minimum of seven replicate reagent water blanks which have been fortified with all analytes of interest at 3 to 5 times the calculated or estimated Instrument Detection Limits (IDLs). IDLs may be calculated by running the calibration blank ten consecutive times. Calculate the standard deviation and multiply by three. If the relative standard deviation from the analysis is less than 10%, the concentration used to determine the MDL is too high. Digest and analyze each replicate according to the procedures described in Section 8.0. Calculate the Method Detection Limit (MDL) by taking the standard deviation of each analyte and multiplying by 3.14. See SOP 110_0013 for a detailed explanation of MDL and also LOD verification.

10.9 The RPD of standards and ICV/CCV should be within ±10% (DOD CCV = ±20%).

11.0 DATA VALIDATION

11.1 Data validation will be accomplished by reviewing all of the quality control parameters and assuring that they are within recommended ranges; any deviations will be recorded in the Graphite Furnace Logbook. The only exceptions made to ranges would be the following:

11.1.1 Analytical batches with BS/BSD outside criteria will be re-prepped and re-analyzed with the following exceptions:

11.1.1.1 If the BS/BSD is biased high, above the acceptance limit, any associated samples that are non-detect may be reported.

11.1.1.2 Non-compliance must be documented in the project narrative.

11.1.2 For duplicates, the RPD should be ± 20% for aqueous samples and ±35% for soil/sediment samples shall be used for sample values greater than 5 times the method reporting limit. Refer to Attachments C and D respectively, for DOD-QSM and MCP-CAM requirements. However, there are cases where duplicates may not work. If this is the case, inform client in narrative concerning sample non-homogeneity.

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11.1.3 For matrix spikes, the %Recovery should be ±25%. Refer to Attachments C and D respectively, for DOD-QSM and MCP-CAM requirements.

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11.1.4 If the matrix spike is outside criteria, check the BS/BSD. If the BS/BSD is within limits, matrix interferences are present and should be noted in the narrative.

11.1.5 Analytical batches with Method blanks above the MRL will be re-prepped and re-analyzed with the following exceptions:

11.1.5.1 Samples that are that are at least twenty times higher than the method blank may be reported.

11.1.5.2 When the method blank is less than 5% of the regulatory limit associated with the analyte the method blank would be acceptable.

11.1.5.3 If the analyte is found in the method blank above the MRL but is not in any of the associated samples, no corrective action is needed.

11.1.5.4 Any results that are reported with method blank contamination must be B-flagged.

11.2 All unusual observations and method deviations will be noted in the narrative accompanying the data report presented to the client.

12.0 REFERENCES

12.1 Methods for the Determination of Metals in Environmental Samples, Supplement I, Supplements to Methods for Chemical Analysis of Water and Wastes, EPA/600/R-94/111 May 1994.

12.2 SW-846, Third Edition, Updates III and IV, 7000 Series methods.

12.3 Standard Methods, 20th Ed. Method 3113B Electrothermal Atomic Absorption Spectrometric Method.

12.4 DOD Quality Systems Manual, Final Version 4.2, October 2010.

12.5 Massachusetts DEP WSC-CAM III C.

13.0 POLLUTION PREVENTION and WASTE MANAGEMENT

13.1 ESS Laboratory's policies on pollution prevention and waste management are covered in SOP 90_0002, Hazardous Waste Contingency and Emergency Response Plan. All employees are trained in the requirements of the SOP.

14.0 DEFINITIONS

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14.1 **Calibration Standard (CAL)** -- A solution prepared from the primary dilution standard solution or stock standard solutions. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

14.2 **Blank spike (BS)** -- An aliquot of reagent water or other blank matrices to which known quantities of the method analytes are added in the laboratory. The BS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.

14.3 **Matrix spike (MS)** -- An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The MS is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS corrected for background concentrations.

14.4 **Method Blank (MB)** -- An aliquot of reagent water or other blank matrices that are treated exactly as a sample including exposure to all glassware, equipment, and reagents that are used with other samples. The MB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

14.5 **Material Safety Data Sheet (MSDS)** -- Written information provided by vendors concerning a chemical's toxicity, health hazards, physical properties, fire, and reactivity data including storage, spill, and handling precautions.

14.6 **Method Detection Limit (MDL)** -- The minimum concentration of an analyte that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero.

14.7 **Quality Control Sample (QCS)** -- A solution of method analytes of known concentrations that is used to fortify an aliquot of BS or sample matrix. The QCS is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check laboratory performance with externally prepared test materials.

14.8 **Stock Standard Solution (SSS)** -- A concentrated solution containing one or more method analytes prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source.

15.0 CORRECTIVE ACTION

The procedure for trouble-shooting and handling out of control situations is described in SOP 80.0009.

16.0 PERSONNEL QUALIFICATIONS

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16.1 BA/BS Chemistry, Natural & Applied Science or equivalent experience.

16.2 All analysts, before performing any analysis, participate in the ESS Laboratory

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training program (SOP80_0016). The training process consists of reading the Standard Operating Procedure, gaining instruction on the procedure from an experienced analyst, and performing the initial demonstration of capability.

17.0 DATA MANAGEMENT AND RECORDS

17.1 ESS Laboratory's utilizes the Promium Element LIMS system as part of its data management system, per SOP 70.0007. The specific retention periods required in the NELAC Standards, EPA-CFR and state and local statutes are followed or exceeded.

18.0 ATTACHMENTS

18.1 Attachment A – Graphite Furnace Run Log.

18.2 Attachment B – Summary of General Method Quality Objectives.

18.3 Attachment C – Summary of DOD Method Quality Objectives.

18.4 Attachment D – Summary of MCP Method Quality Objectives.

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DATE:
ANALYST:
SIF:
RDS:
136 STD 2.0:
139 TI SCV1:
121 STD 2:
124 STD 3:
126 STD 4:
129 STD 5:
131 Recovery STD 50:
134 SCV1:

#	SAMPLE ID	DILUTION	ELEMENTS	#	SAMPLE ID	DILUTION	ELEMENTS
1				37			
2				38			
3				39			
4				40			
5				41			
6				42			
7				43			
8				44			
9				45			
10				46			
11				47			
12				48			
13				49			
14				50			
15				51			
16				52			
17				53			
18				54			
19				55			
20				56			
21				57			
22				58			
23				59			
24				60			
25				61			
26				62			
27				63			
28				64			
29				65			
30				66			
31				67			
32				68			
33				145	Pd Modifier: 1F27041	X5	Ag Cu
34				141	Std 1/CCB: 1G26084		Ag As Cu Pb Se
35				146	NH4 Modifier: 1F23040		Cd Pb
36				147	Pd Modifier: 1F27041		As Sb Se Tl
				148	Std 1/CCB: 1G26084		Cd Sb Tl

Comments:

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ATTACHMENT B

Summary of General Method Quality Objectives for Metals by Graphite Furnace

QC Element	Frequency	Criteria	Corrective Action
Initial Calibration	Daily or each time instrument is set up, prior sample analysis.	• Minimum of calibration blank and three calibration standards. • Low standard at MRL • r≥0.995 (Do not force through zero for LR)	• No allowance. Perform maintenance and recalibrate.
ICV	Immediately following initial calibration.	• %Rec = 90-110%. • Use separate source from initial calibration standards.	• If criteria are exceeded then remake and re-analyze ICV. If second consecutive ICV is acceptable criteria then calibration is accepted, otherwise recalibrate.
CCV	After calibration, every 10 samples and at end of analytical run.	• Concentration level near midpoint of curve • Same source as calibration standard. • %Rec = 90-110% (200.9 first CCV 95-105%)	• If criteria are exceeded then remake and re-analyze CCV. If second consecutive CCV is within criteria then calibration is verified, otherwise re-calibrate system and re-analyze any sample since the last valid CCV.
Continuing Calibration Blank	After calibration, every 10 samples and at end of analytical run.	• Must be matrix-matched (same acid concentration as standards and QC samples.) • Analytes < MRL	• Re-calibrate and re-analyze all samples since last valid CCB.
Method Blank	One per analytical batch of 20 or fewer samples.	• Matrix specific • Analytes < MRL	• Report exceedance in the project narrative. • Any samples that are non-detect for that analyte may be reported. • Samples with concentrations that are 20x higher than the method blank may be reported. • Samples reported with a contaminated blank must be "B" flagged.
Blank spike (LCS)	One per analytical batch of 20 or fewer samples.	• Prepared using standard source different than used for initial calibration • Concentration level should be between low and mid-level standard • Matrix specific • Percent recoveries 80-120%.	• Report exceedance in the project narrative. • If LCS is biased high and sample is non-detect, then may report sample result. • Re-digest and re-analyze if the above exceptions do not apply

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Matrix Spike	One per analytical batch of 10 or fewer samples	• Prepared using the same source as the blank spike • Concentration level should be between low and mid-level standard • Matrix specific • Percent recoveries between 75-125%.	• Report exceedance in the project narrative. • Laboratories are expected to develop in-house control limits per each media. Control limits should fall within default limits.
Matrix duplicate or Sample Duplicate	One per analytical batch of 10 or fewer samples	• **Aqueous**: Relative percent difference is ±20% for samples > 5x MRL and ±MRL for samples <5x the MRL. • **Soil**: Relative percent difference is ±35% for samples > 5x MRL and ±2xMRL for samples <5x the MRL.	• Note exceedance in project narrative.
Serial Dilution	One per analytical batch of 10 or fewer samples, performed on Dup/Spk sample.	• Result of 5x dilution must be ±10% of undiluted sample when result is ≥25x MRL.	• Narrate serial dilution QC out of control. May be indication of matrix affect.
Post Digestion Spike (PDS)	One per analytical batch of 10 or fewer samples, performed on Dup/Spk sample.	• Perform PDS if Serial dilution is not in control or if sample is < 25x the MRL. • Percent recovery must be 85-115%.	• Narrate PDS out of control if MS is also out of control. May be indication of matrix affect.

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Table F-7. Inorganic Analysis by Inductively Coupled Plasma (ICP) Atomic Emission Spectrometry and Atomic Absorption Spectrophotometry (AA) (Methods 6010 and 7000 Series) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial calibration (ICAL) for all analytes **ICP: minimum one high standard and a calibration blank;** **GFAA: minimum three standards and a calibration blank;** **CVAA: minimum 5 standards and a calibration blank**	Daily ICAL prior to sample analysis.	If more than one calibration standard is used, $r \geq 0.995$.	Correct problem, then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed.
Second source calibration verification (ICV)	Once after each ICAL, prior to beginning a sample run.	Value of second source for all analyte(s) within ± 10% of true value.	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
Continuing calibration verification (CCV)	After every 10 field samples and at the end of the analysis sequence.	ICP: within ± 10% of true value; GFAA: within ± 20% of true value; CVAA: within ± 20% of true value.	Correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Low-level calibration check standard (ICP only)	Daily, after one-point ICAL.	Within ± 20% of true value.	Correct problem, then reanalyze.	Flagging criteria are not appropriate.	No samples may be analyzed without a valid low-level calibration check standard. Low-level calibration check standard should be less than or equal to the reporting limit.

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Table F-7. Inorganic Analysis by Inductively Coupled Plasma (ICP) Atomic Emission Spectrometry and Atomic Absorption Spectrophotometry (AA) (Methods 6010 and 7000 Series) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method blank	One per preparatory batch.	No analytes detected > ½ RL and greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common laboratory contaminants, no analytes detected > RL (see Box D-1).	Correct problem, then see criteria in Box D-1. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Calibration blank	Before beginning a sample run, after every 10 samples, and at end of the analysis sequence.	No analytes detected > LOD.	Correct problem. Re-prep and reanalyze calibration blank. All samples following the last acceptable calibration blank must be reanalyzed.	Apply B-flag to all results for specific analyte(s) in all samples associated with the blank.	
Interference check solutions (ICS) (ICP only)	At the beginning of an analytical run.	ICS-A: Absolute value of concentration for all non-spiked analytes < LOD (unless they are a verified trace impurity from one of the spiked analytes); ICS-AB: Within ± 20% of true value.	Terminate analysis; locate and correct problem; reanalyze ICS, reanalyze all samples.	If corrective action fails, apply Q-flag to all results for specific analyte(s) in all samples associated with the ICS.	
LCS containing all analytes to be reported	One per preparatory batch.	QC acceptance criteria specified by DoD, if available; see Box D-3 and Appendix G.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available (see full explanation in Appendix G).	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

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Table F-7. Inorganic Analysis by Inductively Coupled Plasma (ICP) Atomic Emission Spectrometry and Atomic Absorption Spectrophotometry (AA) (Methods 6010 and 7000 Series) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix spike (MS)	One per preparatory batch per matrix (see Box D-7).	For matrix evaluation, use QC acceptance criteria specified by DoD for LCS.	Examine the project-specific DQOs. If the matrix spike falls outside of DoD criteria, additional quality control tests are required to evaluate matrix effects.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix (see Box D-7).	MSD: For matrix evaluation use QC acceptance criteria specified by DoD for LCS. MSD or sample duplicate: RPD ≤ 20% (between MS and MSD or sample and sample duplicate).	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Dilution test (ICP and GFAA only)	One per preparatory batch.	Five-fold dilution must agree within ± 10% of the original measurement.	ICP: Perform post-digestion spike (PDS) addition; GFAA: Perform recovery test.	Flagging criteria are not appropriate.	Only applicable for samples with concentrations > 50 x LOQ.
Post-digestion spike (PDS) addition (ICP only)	When dilution test fails or analyte concentration in all samples < 50 x LOD.	Recovery within 75-125% (see Table B-1).	Run all associated samples in the preparatory batch by method of standard additions (MSA) or see flagging criteria.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	Spike addition should produce a concentration of 1.0 - 100 x LOQ.
Recovery test (GFAA only)	When dilution test fails or analyte concentration in all samples < 25 x LOD.	Recovery within 85-115%.	Run all associated samples in the preparatory batch by method of standard additions (MSA) or see flagging criteria.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	
Method of standard additions (MSA)	When matrix interference is confirmed.	NA.	NA.	NA.	Document use of MSA in the case narrative.
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

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Table F-7. Inorganic Analysis by Inductively Coupled Plasma (ICP) Atomic Emission Spectrometry and Atomic Absorption Spectrophotometry (AA) (Methods 6010 and 7000 Series)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see Section C.1.f).	NA.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification (See Box D-13)					
LOQ establishment and verification (See Box D-14)					
Instrument detection limit (IDL) study (ICP only)	At initial set-up and after significant change in instrument type, personnel, test method, or sample matrix.	IDLs shall be ≤ LOD.	NA.	NA.	Samples may not be analyzed without a valid IDL.
Linear dynamic range or high-level check standard (ICP only)	Every 6 months.	Within ± 10% of true value.	NA.	NA.	

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Table III C-1: Specific QC Requirements and Performance Standards for Metals (SW-846 7010) Using WSC-CAM-III C

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
Initial Demonstration of Proficiency (IDP)	Laboratory Analytical Accuracy & Precision	(1) Must be performed prior to using method on samples. (2) Must be performed for each matrix. (3) Must contain all target analytes. (4) Must follow procedures in Section 9.4 of SW-846 7010 and the applicable preparation method (SW-846 3000 series).	No	NA	Refer to Section 9.4 of SW-846 7010, the applicable preparation method requirements in SW-846 3000 series methods, and Section 1.1.2 of this protocol.	NA
Preparation of Samples	Accuracy and Representativeness	(1) All aqueous (except dissolved/filtered groundwaters) and solid samples must be prepared (digested) prior to analysis. See Appendix III C-4 for preparation method references.	No	NA	NA	NA
Initial Calibration	Laboratory Analytical Accuracy	(1) Frequency: Daily prior to sample analysis. (2) Minimum calibration blank plus 3 calibration standards (multi-point) which may include the RL (LLCV) standard; if LLCV standard is not included in calibration curve, then LLCV QC sample is required (see below). High level standard in calibration defines the upper end of the linear calibration range. (3) Linear regression with correlation coefficient r $\geq$0.995.	No	NA	Perform instrument maintenance as necessary; re-optimize instrument; re-calibrate as required by SW-846 7010.	Suspend all analyses until initial calibration meets criteria.
Initial Calibration Verification (ICV)	Laboratory Analytical Accuracy	(1) Frequency: Immediately after each initial calibration. (2) Prepared using standard source different than used for initial calibration. (3) Concentration level near midpoint of curve. (4) Must contain all target analytes. (5) Percent recoveries must be between 90-110% for each target analyte.	No	NA	(1) Reanalyze ICV; if acceptable, no further action required. (2) If reanalysis is still outside of criteria, recalibrate and reanalyze ICV.	Suspend all analyses until ICV meets criteria.

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Table III C-1: Specific QC Requirements and Performance Standards for Metals (SW-846 7010) Using WSC-CAM-III C

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
Initial Calibration Blank (ICB)	Laboratory Analytical Sensitivity (instrument drift & contamination)	(1) Frequency: Immediately after ICV. (2) Prepared using same concentration of acids as calibration standards. (3) Target analytes must be <RL.	No	NA	(1) Reanalyze ICB; if acceptable, no further action required. (2) If reanalysis is still outside of criteria, recalibrate and reanalyze ICV & ICB.	Suspend all analyses until ICB meets criteria.
Low-Level Calibration Verification (LLCV)	Laboratory Analytical Sensitivity (verify low-end of calibration range / verify RL)	(1) Frequency: Daily prior to sample analysis if initial calibration did not contain a low-level standard at the RL. If initial calibration includes the RL as the low-level standard in the initial calibration curve, then LLCV is not required. (2) Prepared using same source as initial calibration standards. (3) Concentration level must be at the level of the RL for all target analytes. (4) Percent recoveries must be 70-130% for all target analytes.	No	NA	(1) Reanalyze LLCV; if acceptable, no further action required. (2) If reanalysis is still outside of criteria and associated analytes are ≤10x RL in associated field samples, recalibrate and reanalyze LLCV and associated samples. (3) If associated analytes are >10x RL in associated field samples, include explanation in laboratory narrative; no further action required.	Suspend all analyses until LLCV meets criteria unless the concentrations of the affected target analytes are >10x RL in the associated field samples.
Continuing Calibration Verification (CCV)	Laboratory Analytical Accuracy	(1) Frequency: Every 10 samples and at the end of the analytical run. (2) Prepared using same source as initial calibration standards. (3) Concentration level near midpoint of curve. (4) Must contain all target analytes. (5) Percent recoveries must be 90-110% for each target analyte.	No	NA	(1) Reanalyze CCV; if acceptable, no further action required. (2) If reanalysis is still outside of criteria, recalibrate and reanalyze all associated samples since last compliant CCV – unless (3) applies. (3) If recovery is high (>110%) and all associated sample results are non-detected, no corrective action required	If (3) applies, include explanation in laboratory narrative.

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Table III C-1: Specific QC Requirements and Performance Standards for Metals (SW-846 7010) Using WSC-CAM-III C

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
Continuing Calibration Blank (CCB)	Laboratory Analytical Sensitivity (instrument drift & contamination)	(1) Frequency: Every 10 samples following CCV and at the end of the analytical run. (2) Prepared using same concentration of acids as calibration standards. (3) Target analytes must be <RL.	No	NA	(1) Reanalyze CCB; if acceptable, no further action required. (2) If reanalysis is still outside of criteria, recalibrate and reanalyze all associated samples since last compliant CCB – unless (3) applies. (3) If concentration of contaminant in CCB is >RL but all associated sample results are either non-detected or >10x concentration of contaminant in CCB; no corrective action required.	If (3) applies, include explanation in laboratory narrative.
Method Blank (MB)	Laboratory Method Sensitivity (contamination evaluation)	(1) Frequency: One per digestion batch of ≤20 field samples. (2) Must be digested with the samples using the same preparation method as the samples. (3) Target analytes must be <RL.	Yes	NA	(1) Reanalyze MB; if acceptable, no further action required. (2) If reanalysis is still outside of criteria, redigest and reanalyze MB and all associated field samples in batch – unless (3) applies. (3) If concentration of contaminant in MB is >RL but all associated sample results are either non-detected or >10x concentration in MB; no corrective action required.	If (3) applies, include explanation in laboratory narrative.

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Table III C-1: Specific QC Requirements and Performance Standards for Metals (SW-846 7010) Using WSC-CAM-III C

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
Laboratory Control Sample (LCS)	Laboratory Analytical Accuracy	(1) Frequency: One per digestion batch of ≤20 field samples. (2) Must be matrix-matched by digesting with the samples using the same preparation method. CAM requires a solid Standard Reference Material (SRM) be prepared and analyzed with solid field samples as the "solid LCS." An SRM is a soil or sediment matrix that contains the analytes of interest at known concentrations and with 95% confidence limits. (3) Concentration levels for aqueous LCS near midpoint of curve. (4) Must contain all target analytes. (5) Percent recoveries for all target analytes must be 80-120% for aqueous LCS and within vendor control limits (95% confidence limits) for solid LCS.	Yes	Aqueous LCS: Recovery <50%: affected analytes in associated samples may be rejected.	(1) Reanalyze LCS; if acceptable, no further action required. (2) If reanalysis is still outside of criteria and LCSD is in-control for same analyte; no corrective action required. (3) If LCS and LCSD are both outside of criteria, redigest and reanalyze LCS/LCSD and all associated field samples in batch.	Report recovery exceedances in laboratory narrative.

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WSC-CAM-III C

Table III C-1: Specific QC Requirements and Performance Standards for Metals (SW-846 7010) Using WSC-CAM-III C

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
LCS Duplicate (LCSD)	Laboratory Analytical Accuracy & Precision	(1) Frequency: One per digestion batch of ≤20 field samples ONLY if not performing project-specific MD. (2) Must be matrix-matched by digesting with the samples using the same preparation method. CAM requires a solid SRM be prepared and analyzed with solid field samples as the "solid LCSD." An SRM is a soil or sediment matrix that contains the analytes of interest at known concentrations and with 95% confidence limits. (3) Concentration levels must be same as LCS. (4) Must contain all target analytes; analyze immediately following LCS. (5) Percent recoveries for all target analytes must be 80-120% for aqueous LCS and within vendor control limits (95% confidence limits) for solid LCS. (6) RPDs must be ≤20 for aqueous LCS/LCSD and ≤30 for solid LCS/LCSD.	Yes ONLY if no MD	Same as above for LCS for recovery evaluation	(1) Reanalyze LCSD; if acceptable, no further action required. (2) If reanalysis is still outside of recovery criteria and LCS is in-control for same analyte, no corrective action required. (3) If LCSD and LCS are both outside of recovery criteria, redigest and reanalyze LCS/LCSD and all associated field samples in batch.	Report recovery and RPD exceedances in laboratory narrative.
Matrix Spike (MS) Project-Specific	Method Accuracy in Sample Matrix	(1) Solid Samples (Soil/Sediment) Frequency: One per 20 field samples per matrix; designated by data user on COC or at project set-up. Aqueous Samples Frequency: One per digestion batch of ≤20 field samples per matrix strongly recommended (designated by data user on COC or at project set-up). (2) Concentration levels near midpoint of curve. (3) Must contain all target analytes. (4) Percent recoveries for all target analytes must be 75-125%.	Yes ONLY when requested by the data user	Recovery <30%: affects non-detects for affected metal in all associated samples.	(1) Reanalyze MS; if acceptable, no further action required. (2) After reanalysis, if MS recovery is 30-74% or >125% and LCS was in-control, no corrective action is required. (3) If MS recovery is <30% and associated with non-detected results, redigest (homogenize sample well) and reanalyze sample/MS pair. Report results and narrate.	Report MS exceedances in laboratory narrative. If redigested due to recoveries <30%, report both sets of sample/MS data.

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Table III C-1: Specific QC Requirements and Performance Standards for Metals (SW-846 7010) Using WSC-CAM-III C

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
Matrix Duplicate (MD) Project-Specific	Method Precision in Sample Matrix	(1) Frequency: One per digestion batch of ≤20 field samples per matrix is strongly recommended (designated by data user on COC or at project set-up). (2) Prepare by digesting and analyzing an additional aliquot of the same field sample used for MS. (3) RPD for each target analyte must be ≤20 for aqueous and ≤35 for solids.	Yes ONLY when requested by the data user	NA	Narrate.	Report exceedances in laboratory narrative.
Dilution Test	Accuracy in Sample Matrix	(1) Frequency: One per ≤20 field samples per matrix; only if project-specific MS requested and analyte concentration is >50x RL. (2) Perform 5x serial dilution on same sample used for MS/MD. (3) %D of the Sample & Dilution results for target analytes at levels >50x RL must be ±10% for all matrices.	Yes ONLY if project-specific MS requested by data user	NA	Narrate.	Report exceedances in laboratory narrative.
Duplicate Injections	Method Precision	(1) Frequency: Each calibration standard, QC sample, and field sample must be analyzed (injected) twice. (2) RPD must be ≤10 for calibration standards and ≤20 for all other detected results. (3) Report the average result of duplicate injections for all target metals.	No	NA	(1) Reanalyze; if duplicate injection RPD meets criteria, no further action required. (2) If RPD still outside of criteria, dilute sample and re-analyze diluted sample with duplicate injections. (3) If RPD still outside of criteria on dilution, narrate.	Report Duplicate Injection RPD exceedances in laboratory narrative – potential sample matrix interference.

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Division of Thielsch Engineering
Cranston, RI

SOP NO 30_2451
MERCURY ANALYSIS FOR AQUEOUS SAMPLES
(METHODS EPA 245.1/ SW846 7470A)

REVIEWED BY:

Operations Manager	Date 5/3/05
QA Manager	Date 4/19/05
Laboratory Director	Date 4/27/05

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MERCURY ANALYSIS FOR AQUEOUS SAMPLES (METHODS EPA 245.1 / SW-846 7470A)

1.0 SCOPE AND APPLICATION

1.1 This method measures the total mercury content in prepared aqueous samples (drinking, waste, surface, and ground waters), TCLP extracts, and wastes that contain suspended solids by cold vapor atomic absorption. This method can determine mercury concentrations as low as 0.5 ppb and as high as percent levels, with the applicable dilution.

2.0 SUMMARY OF METHOD

2.1 In a 50-ml centrifuge tube, a 20-ml portion of an aqueous sample or extract is digested in the presence of nitric acid, sulfuric acid, potassium permanganate, and potassium persulfate. After digestion, sodium chloride-hydroxylamine hydrochloride, and stannous chloride are added, thereby releasing the mercury as a vapor. The mercury vapor is aerated into a closed cell positioned in the light path of an atomic absorption spectrophotometer capable of measuring radiation at 253.7 nm.

3.0 HEALTH AND SAFETY

3.1 Each employee has been trained and has acknowledged being trained in the safe use and handling of chemicals being used in the laboratory. This training has been performed according to the ESS Training SOP 80_0016 and by the Chemical Hygiene Plan, SOP No. 90_0001, in conjunction with the Safety orientation.

3.2 All sample and material handling is performed in a hood while using proper protective equipment to minimize exposure to liquid or vapor. Minimum personnel protective equipment includes the use of laboratory safety glasses, a lab coat or apron, and protective gloves.

3.3 The MSDS for the concentrated chemicals used in the laboratory are kept on file in a central location that is available for all employees to review.

3.4 Proper emergency response to spills or injury should be reviewed by the laboratory employee prior to attempting this procedure. Employees must know the location of spill kits, eyewashes, showers, and fire fighting equipment. Employees must also have knowledge of disaster evacuation routes.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

4.1 Prior to analysis, all samples are to be preserved with HNO_3 to a pH of less than two. If upon receipt the pH requires adjusting, nitric acid is added to the sample until the pH has stabilized. The metal laboratory will then hold the sample for 16 hours.

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4.2 Hold time for aqueous samples is 28 days from day of sampling. Samples prepared after this date are flagged as estimated values.

4.3 For dissolved metals analysis, a non-preserved sample must be filtered through a 0.45 µm filter within 24 hours of collection and preserved to a pH of less than two.

4.4 Immediately prior to preparation, an aliquot of the sample is checked to insure that the pH is less than 2. If the pH is greater than 2, additional acid is added, the pH is retested, and the sample is held 16 hours and the above process is repeated until pH is verified to be <2. The total volume of acid added must not be greater than 1% of the sample volume. If the pH > 2 after 1% acid has been added to the sample, then the exceedance is noted in the project narrative.

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

5.1 Potassium persulfate is added to eliminate possible interference from sulfide.

5.2 High chlorides such as those found in salt-water samples require additional potassium permanganate to convert the chlorides to free chlorine. The addition of solid potassium permanganate is often employed. A reagent blank should be treated similarly. Because free chlorine also absorbs radiation at 253.7 nm, care must be taken to ensure that all the free chlorine is absent before the mercury is reduced and swept into the cell. This is accomplished by using an excess of hydroxylamine hydrochloride as well as purging the headspace in the centrifuge tube prior to adding the stannous chloride.

5.3 Certain volatile organic materials that absorb at this wavelength may also cause interference. A preliminary run without reagents should determine if this type of interference exists.

6.0 EQUIPMENT/APPARATUS

6.1 Perkin Elmer **Flow Injection Mercury System, FIAS 100** (serial # B0509550).

6.2 Perkin Elmer **AS90 Autosampler** (serial#3353).

6.3 50 ml **graduated centrifuge tubes**.

6.4 **Hot Block**, digester by Environmental Express

6.5 **Disposable screw cap covers**, by Environmental Express

6.6 **0.2 micron FilterMates**, by Environmental Express

6.7 **Volumetric Flasks**: 200 ml, 250 ml, and 1L.

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6.8 **Pipettes**: Class A or calibrated pipettors.

6.9 Wide range pH paper

6.10 Disposable **Pasteur pipettes**

7.0 REAGENTS

7.1 **Reagents**: All reagents used must be trace-metal grade and interference free.

7.1.1 **Concentrated Hydrochloric Acid** (HCl): The method blank must be less than ½ the MRL in order to use.

7.1.1.1 **3% Hydrochloric Acid**: In the designated carboy, add 600 ml of concentrated HCl to approximately 10L of DI water. Volumize to 20 L mark, cap and shake carboy carefully to mix. Smaller volumes may be prepared.

7.1.2 **Concentrated Sulfuric Acid** (H_2SO_4): The method blank must be less than ½ the MRL in order to use.

7.1.3 **Concentrated Nitric Acid** (HNO_3): The method blank must be less than ½ the MRL in order to use.

7.1.4 **Potassium Permanganate** ($KMnO_4$), 5% **solution**: In a 1L volumetric flask, add 50g of Potassium Permanganate and dilute to 1L mark with DI water. This is a likely source of mercury contamination. The potassium permanganate used should be a "mercury free" grade.

7.1.5 **Potassium Persulfate** ($K_2S_2O_8$), 5% **solution**: In a 1L volumetric flask, add 50g of potassium persulfate and dilute to 1L mark with DI water.

7.1.6 **Sodium Chloride-Hydroxylamine Hydrochloride** ($NH_2OH{\cdot}HCl$) **solution**: In a 1L volumetric flask, add 120g of hydroxylamine hydrochloride and 120g of sodium chloride and dilute to 1L mark with DI water.

7.1.7 **Stannous Chloride solution**: In a 1L volumetric flask, add 11 g of Stannous Chloride, to approximately 500 ml of 3% HCl (7.1.1.1). Volumize to 1L with 3% HCl.

7.2 **Standards**:

7.2.1 **Primary Standards**: Stored at room temperature and not used beyond the manufacturer's expiration date.

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Vendor	catalog #	Concentration
Aldrich	20,729-2	1000 ug/ml
CPI	4400-1000331	1000 ug/ml

7.2.2 **Working Calibration Standard Solutions** are prepared daily:

7.2.2.1 **Hg Stock Standard (1 ppm):** In a 250 ml volumetric flask, add 100 ml of DI water, 7.5 ml of HNO_3, and 0.25 ml of the CPI standard (see 7.2.1). Volumize to the 250 ml mark with DI water. ***This solution is stable for one week.***

7.2.2.2 **HgICV/BS/BSD/MS/MSD Stock Standard (1ppm):** In a 250 ml volumetric flask, add 100 ml DI water, 7.5 ml HNO_3, and 0.25 ml of the Aldrich standard (see 7.2.1). Volumize to the 250 ml mark with DI water. ***This solution is stable for one week.***

7.2.2.3 **Calibration Standards** are used to calibrate the instrument. Prepared in labeled centrifuge tubes using the Hg stock standard (see 7.2.2.1), with 20 ml of DI water (proceed to section 8). Treat the standards as samples without the heating step.

centrifuge tube label	volume of standard	initial volume	final concentration[1]
0.0 ppb	0 ml	20 ml	0 ppb
0.25 ppb	0.01 ml	20 ml	0.25 ppb
0.5 ppb	0.02 ml	20 ml	0.5 ppb
1.0 ppb	0.04 ml	20 ml	1.0 ppb
3.0 ppb	0.12 ml	20 ml	3.0 ppb
5.0 ppb	0.2 ml	20 ml	5.0 ppb

[1] Based on a final volume of 40 ml.

7.2.2.4 The **Instrument Check Standards** (ICV and CCV: Initial and Continuing Calibration Verification) are used to verify the instrument calibration.

7.2.2.4.1 The **Continuing Calibration Verification** (CCV) is analyzed immediately after the curve, after every ten samples, and at the end of the run. The CCV is prepared by pipetting 0.12 ml of Hg stock (see 7.2.2.1) in a centrifuge tube and diluting to 20 ml with DI water. Proceed to section 8. Treat standards as samples, without the heating step.

7.2.2.4.2 The **Initial Calibration Verification** (ICV) is analyzed immediately after the initial CCV. The ICV is prepared by pipetting 0.12 ml of HgICV/LCS/MS stock standard (see 7.2.2.2) in a centrifuge tube and diluting to 20 ml with DI

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water. Proceed to section 8. Treat standards as samples, without the heating step.

7.2.2.5 Every batch of samples prepped must have a **Blank Spike (BS) and Blank Spike Duplicate (BSD)**. Label two tubes with the BS and BSD IDs, pipette 0.12 ml of HgICV/BS/BSD/MS/MSD stock standard (see 7.2.2.2) and volumize to 20 ml with DI water. Prep the BS and BSD as samples (see section 8).

7.2.2.6 Every batch of 10 samples must have a **matrix spike (MS) and matrix spike duplicate (MSD)**. Prepare MS and MSD by adding 20 ml of sample to two labeled centrifuge tubes. Pipette 0.12 ml of HgICV/BS/BSD/MS/MSD stock standard (see 7.2.2.2) into each tube. Prep the MS and MSD as samples (see section 8).

7.3 **Three types of blanks** are required for the analysis. The **calibration blank** is used in establishing the analytical curve, the **method blank** is used to assess possible contamination resulting from the preparation step, and the **continuing calibration blank** (CCB) verifies the calibration blank as well as check for instrument contamination.

7.3.1 The calibration blank (ICCB) and continuing calibration blank are prepared by adding 20 ml of DI water to a centrifuge tube. Proceed to section 8. Treat as a sample without the heating step.

7.3.2 The method blank is prepared by filling a centrifuge tube with 20 ml of DI water. The method blank must be carried through the complete procedure and contain the same acid and reagent concentrations as the samples (section 8).

8.0 PROCEDURE

8.1 **Sample Digestion**:

8.1.1 Take the pH of each sample using pH paper. Record the pH in the Mercury prep logbook (Attachment A). If the pH >2, see section 4.0 for corrective action.

8.1.2 Add 20 ml of sample, or an aliquot diluted to 20 ml with DI water, to a centrifuge tube. Record volume of sample on Hg prep log sheet (Attachment A), or electronic device (HP Tablet), as described in Attachment E.

8.1.3 Add 1.0 ml of concentrated sulfuric acid to each sample and mix.

8.1.4 Add 0.5 ml of concentrated nitric acid to each sample and mix.

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8.1.5 Add 3.0 ml of potassium permanganate solution to each sample and mix. If liquid does not turn or remain purple, a dilution of the sample is required, or an increased amount of reagent should be added. Ensure that equal amounts of reagent are added to standards and blank. At the end of the two-hour digestion the sample must remain purple.

8.1.6 Add 1.6 ml of potassium persulfate solution to each sample and mix.

8.1.7 Cap each tube.

8.1.8 Place in Hot Block at 95°C for two hours and record both start/end times and temperature.

8.1.9 Allow samples to cool to room temperature.

8.1.10 Add 1 ml of sodium chloride hydroxylamine hydrochloride to each sample and standard. At the end of this step, the solution should be clear (no pink or purple present). ***NOTE: Samples must be analyzed as soon as possible after decolorization.***

8.1.11 Volumize each sample and standard to 40 ml with DI water and mix.

8.1.12 Transfer samples from centrifuge tubes to autosampler tubes and record in Hg run log (Attachment B), or HP Tablet, as described in Attachment E.

8.2 **Sample Analysis**:

8.2.1 Instrument, Computer, Argon tank, and printer should be turned on.

8.2.2 Fill instrument bottles with stannous chloride solution and 3% HCl (see 7.1.1.1 and 7.1.7). Lock pump tension bars in place. Replace tubing if necessary.

8.2.3 Using Perkin Elmer WinLab software, create a sample information file from the mercury run log, and print the auto sample load list. Place samples on autosampler using the auto sample load list as a guide.

8.2.4 Select the auto method Hg-5ppb Aq, and create a data results file where data will be saved.

8.2.5 Select print log to print run data, and if running unattended, select turn off lamp at end of run.

8.2.6 Start analytical run.

9.0 CALCULATIONS

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9.1 The results are given in µg/L. Final results are converted to mg/L (by dividing by 1000) and then multiplying by a final volume of 40 ml and dividing by the initial volume of sample.

9.2 Before data is submitted for reporting, sample ID's and units are checked for transcription errors.

10.0 QUALITY ASSURANCE/QUALITY CONTROL

10.1 All quality control data should be maintained and available for easy reference or inspection.

10.2 Dilute and reanalyze samples that are more concentrated than the highest standard.

10.3 The initial calibration consists of a blank and five standards. The coefficient of determination (R^2) for the standard curve must be greater than or equal to 0.995.

10.4 Initial and Periodic Method QC Demonstrations: The procedures specified in Section 10.4.1 through 10.4.2 must be conducted as an initial demonstration of laboratory capability, prior to the analysis of any samples. Subsequent to this initial demonstration, additional evaluations of this nature should be conducted on a periodic basis, in response to changes in instrumentation or operations, and/or in response to confirmed or suspected systems, method, or operational problems.

10.4.1 Accuracy and Precision: To demonstrate initial laboratory capability, analyze a minimum of four initial calibration verification standards. The mean concentration must be within ± 10 % of the stated values.

10.4.2 A MDL must be established using a blank fortified at a concentration of two to three times the estimated detection limit. Seven replicate aliquots are processed through the entire method. See SOP 110_0013 for a detailed explanation.

10.5 The CCV and CCB must be analyzed immediately after the curve, after every tenth sample, and at the end of the sample run.

10.5.1 The CCV that is run immediately after the curve must have a recovery within the range of 95-105% (80-120% for DOD and MCP).

10.5.2 The CCV that is run after every tenth sample and at the end of the sample run must have a recovery range of 80-120%.

10.5.3 The value for the CCB must not exceed ½ the MRL (DOD requires ≤½ LOD).

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10.5.4 If either the CCV or CCB are invalid, analyze all samples analyzed since the last valid CCV/CCB

10.6 The ICV must be analyzed immediately following the initial calibration to verify the validity of the primary standard.

10.6.1 The ICV recovery range must be within the range of 90-110%.

10.6.2 If the ICV is outside of this range, a new daily calibration must be generated.

10.7 Employ a minimum of one method blank per sample batch (20 samples maximum) to determine if contamination is occurring. The method blank must contain all the reagents and in the same volumes as used in the processing of the samples. It must be carried through the complete procedure as the sample analysis.

10.7.1 The value for the blank should not exceed ½ the MRL (see Attachment C for DOD requirements). See Section 11.0 for corrective action on method blanks.

10.8 On an ongoing basis the laboratory analyzes a matrix spike for every ten samples or per analytical batch, whichever is more frequent. Calculate the percent recovery as follows:

$$\% \text{ Recovery} = \frac{SSR - SR}{SA} \times 100$$

Where:

SSR = Spiked Sample Result
SR = Sample Result
SA = Spike Added to Sample

10.8.1 The control limit is 75-125% for %Recovery when sample concentration is less than four times the amount of spike added. (***For DOD work the control limit is 80-120% Recovery.***)

10.9 Matrix duplicate samples will only be analyzed at the client's request. Calculate the relative percent difference as follows:

$$RPD = \frac{D_1 - D_2}{(D_1 + D_2)/2} \times 100$$

Where:

RPD = relative percent difference
D_1 = first sample value
D_2 = second sample value (replicate)

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10.9.1 The control limit is ±20% for RPD for samples that are >5 x MRL and ± the MRL for samples that are ≤ MRL. If the samples fall outside the control limit, see section 11.0

10.10 At least one Blank spike and Blank spike duplicate must be analyzed for each batch of samples. The BS and BSD are prepared and analyzed in the same manner as the samples.

10.10.1 The BS/BSD recovery must be within the range of 80-120% (85-115% for method 245.1 and have an RPD ≤ 20%.

10.10.2 If the BS or BSD is outside criteria, then re-prep and re-analyze samples with the following exception: ***For high LCS, those samples that are non-detects may be reported.***

10.11 Any deviations from methodology or unusual observations will be noted in the narrative that accompanies data.

10.12 Control charts will be generated through Element LIMS for method blanks and LCSs to determine if in-house limits fall within SOP acceptance criteria.

10.13 The quantitation limit (QL) for this method is verified by running a low level calibration verification. Recovery requirement is 70-130% (80-120 for DOD). The method reporting limit is calculated as follows:

$$MRL = \frac{QL \times \text{Final volume (ml)} \times \text{dilution factor}}{\text{Initial volume (ml)}}$$

10.14 Refer to Attachment D for a summary of MCP method quality objectives.

11.0 DATA VALIDATION

11.1 Data validation will be accomplished by reviewing all of the quality control parameters and assuring that they are within recommended ranges. Any deviations will be recorded in the run log. The only exceptions made to ranges would be the following:

11.1.1 For duplicates, the RPD should be ± 20%. However, there are cases where duplicates may not work. If this is the case, inform client in narrative concerning sample non-homogeneity.

11.1.2 For matrix spikes, the % Recovery should be ±25% (***±20% for DOD work***). If the matrix spike is outside acceptance criterion, check the LCS. If the LCS is within limits, matrix interferences are present and should be noted in the narrative.

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11.1.3 Analytical batches with Method blanks above the MRL (<LOD for DOD) will be re-prepped and re-analyzed with the following exceptions:

11.1.3.1 Samples that are at least twenty times higher than the method blank may be reported.

11.1.3.2 When the method blank is less than 5% of the regulatory limit associated with the analyte, the method blank would be acceptable.

11.1.3.3 If the analyte is found in the method blank above the MRL but is not in any of the associated samples, no corrective action is needed.

11.1.3.4 Any results that are reported with method blank contamination must be B-flagged.

11.2 All unusual observations and method deviations will be noted in the narrative accompanying the data report presented to the client.

12.0 REFERENCES

12.1 U.S. Environmental Protection Agency, Methods for the Determination of Metals in Environmental Samples, Supplement 1, EPA-600/R-94/111. May 1994. Method 245.1

12.2 SW846 Method 7470A: Mercury in Liquid Waste (Manual Cold-Vapor Technique). Third edition, Updates III and IV.

12.3 FIMS, Flow Injection Mercury System, Perkin-Elmer Part # 0093-5203

12.4 DOD Quality Systems Manual, Final Version 4.2, October 2011.

12.5 Massachusetts DEP WSC-CAM III B.

13.0 POLLUTION PREVENTION and WASTE MANAGEMENT

13.1 ESS Laboratory's policies on pollution prevention and waste management are covered in SOP 90_0002, Hazardous Waste Contingency and Emergency Response Plan. All employees are trained in the requirements of the SOP.

14.0 DEFINITIONS

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14.1 **Calibration Blank (CB)** -- A volume of reagent water fortified with the same matrix as the calibration standards, but without the analytes.

14.2 **Calibration Standard (CAL)** -- A solution prepared from the primary dilution standard solution or stock standard solutions. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

14.3 **Blank spike (BS)** -- An aliquot of reagent water or other blank matrices to which known quantities of the method analytes are added in the laboratory. The BS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.

14.4 **Matrix spike (MS)** -- An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The MS is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS corrected for background concentrations.

14.5 **Method Blank (MB)** -- An aliquot of reagent water or other blank matrices that are treated exactly as a sample including exposure to all glassware, equipment, and reagents that are used with other samples. The MB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

14.6 **Material Safety Data Sheet (MSDS)** -- Written information provided by vendors concerning a chemical's toxicity, health hazards, physical properties, fire, and reactivity data including storage, spill, and handling precautions.

14.7 **Method Detection Limit (MDL)** -- The minimum concentration of an analyte that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero.

14.8 **Quality Control Sample (QCS)** -- A solution of method analytes of known concentrations that is used to fortify an aliquot of BS or sample matrix. The QCS is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check laboratory performance with externally prepared test materials.

14.9 Stock Standard Solution (SSS) -- A concentrated solution containing one or more method analytes prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source.

15.0 METHOD PERFORMANCE

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15.1 Precision and Accuracy data must be generated by all employees before performing this analysis on client samples. The data is generated by analyzing a method blank and four blank spike samples. Acceptance criteria are 85-115% Recovery and %RSD of ≤ 20%.

15.2 The precision and accuracy data in Table 1 were developed using a reagent water matrix. Values are in mg/L.

16.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

Table 1. Typical Precision and Accuracy data generate

Analyte	Date	True value	Average	%Recovery	%RSD
Mercury	2/3/05	3.0	2.79	93	3

17.0 PERSONNEL QUALIFICATIONS

17.1 BA/BS Chemistry, Natural & Applied Science or equivalent experience.

17.2 All analysts, before performing any analysis, participate in the ESS Laboratory training program (SOP80_0016). The training process consists of reading the Standard Operating Procedure, gaining instruction on the procedure from an experienced analyst, and performing the initial demonstration of capability.

18.0 TROUBLESHOOTING/MAINTENANCE

18.1 Change tubes, membranes and filters as needed.

18.2 If necessary, contact Perkin-Elmer for service.

19.0 DATA MANAGEMENT AND RECORDS

19.1 ESS Laboratory's utilizes the Promium Element LIMS system as part of its data management system, per SOP 70.0007. The specific retention periods required in the NELAC Standard, EPA-CFR and state and local statutes are followed or exceeded.

20.0 ATTACHMENTS (Including TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA)

20.1 Attachment A – Prep Logbook

20.2 Attachment B – Run logbook

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20.3 Attachment C – General and DOD Method Quality Objectives.

20.4 Attachment D -- MCP Method Quality Objectives.

20.5 Attachment E -- Procedure for recording sample information electronically.

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Attachment A

Analyst:

Date:

Batch ID:

Reagent IDs:

H_2SO_4 | $K_2S_2O_8$

HNO_3 | NaCl-NH_2OH*HCl

$KMnO_4$ | Cal/Spk Std ID*:

ICV std ID**:

Sample ID #	pH	Sample Init Vol (ml	Cal/ ICV/ Spk Vol (ml)	Comments	Final Vol (ml)	Bath #	Temp (°C)	Time in	Time out

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Attachment B

SIF:

$SnCl_2 \cdot 2H_2O$ ID:

RDS:

CARRIER ID:

ANALYST:

DATE:

Matrix: Solid / Aqueous

FIMS AUTOSAMPLER TRAY SEQUENCE

#	SAMPLE	#	SAMPLE	#	SAMPLE	#	SAMPLE
1	Std 1/CCB	31		61		91	
2	Std 2-	32		62		92	
3	Std 3-	33		63		93	
4	Std 4 -	34		64		94	
5	Std 5-	35		65		95	
6	Std 6-	36		66		96	
7	ICV-	37		67		97	
8		38		68		98	
9		39		69		99	
10		40		70		100	
11		41		71		101	
12		42		72		102	
13		43		73		103	
14		44		74		104	
15		45		75		105	
16		46		76		106	
17		47		77			
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22		52		82			
23		53		83			
24		54		84			
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Attachment C
Summary of Method Quality Objectives for Method 245.1/7470A and DOD Mercury by CVAAS

QC Element	Frequency	Criteria	Corrective Action
Initial Calibration	Daily or each time instrument is set up, prior sample analysis.	• Minimum of calibration blank and five calibration standards. • r≥0.995 (Do not force through zero for LR)	• No allowance. Perform maintenance and recalibrate.
ICV	Immediately following initial calibration.	• %Rec = 90-110%. • Use separate source from initial calibration standards.	• If criteria are exceeded then remake and re-analyze ICV. If second consecutive ICV is acceptable criteria then calibration is accepted, otherwise recalibrate.
CCV	After calibration, every 10 samples and at end of analytical run.	• Concentration level near midpoint of curve • Same source as calibration standard. • %Rec = 90-110% (200.9 first CCV 95-105%) • DOD QSM %Recovery = 80-120%	• If criteria are exceeded then remake and re-analyze CCV. If second consecutive CCV is within criteria then calibration is verified, otherwise re-calibrate system and re-analyze any sample since the last valid CCV.
Continuing Calibration Blank	After calibration, at start of run, every 10 samples and at end of analytical run.	• Must be matrix-matched (same acid concentration as standards and QC samples.) • Analytes < MRL DOD specifies Analytes < LOD	• Re-calibrate and re-analyze all samples since last valid CCB.
Method Blank	One per analytical batch of 20 or fewer samples.	• Analytes < MRL • DOD specifies Analytes < ½ MRL and < 1/10 sample concentration or regulatory limit (whichever is greater)	• Report exceedance in the project narrative. • Any samples that are non-detect for that analyte may be reported. • Samples with concentrations that are 20x higher than the method blank may be reported. • Samples reported with a contaminated blank must be "B" flagged.
Blank spike (LCS)	One per analytical batch of 20 or fewer samples.	• Prepared using standard source different than used for initial calibration	• Report exceedance in the project narrative. • If LCS is biased high and sample is non-

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		• Concentration level should be between low and mid-level standard • Percent recoveries 80-120%.(85-115% for 245.1)	detect, then may report sample result. • Re-digest and re-analyze if the above exceptions do not apply.
Matrix Spike	One per analytical batch of 10 or fewer samples	• Prepared using the same source as the blank spike • Concentration level should be between low and mid-level standard • Matrix specific • Percent recoveries between 75-125%. • ***DOD 80-120%.***	• Report exceedance in the project narrative. • Laboratories are expected to develop in-house control limits per each media. Control limits should fall within default limits.
Matrix duplicate	At client's request.	• Relative percent difference is $\pm$20% for samples > 5x MRL and $\pm$MRL for samples < 5x the MRL.	• Note exceedance in project narrative.
Results reported between DL and LOQ (MRL)	NA	NA	Apply J-Flag

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Table III B-1: Specific QC Requirements and Performance Standards for Mercury (SW-846 7470A & 7471B) Using WSC-CAM-III B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
Initial Demonstration of Proficiency (IDP)	Laboratory Analytical Accuracy & Precision	(1) Must be performed prior to using method on samples. (2) Must be performed for each matrix. (3) Must follow procedures in Section 9.2 of SW-846 7471B and Section 9.4 of SW-846 7000B.	No	NA	Refer to Section 9.2 of SW-846 7471B, Section 9.4 of SW-846 7000B, and Section 1.1.2 of this protocol.	NA
Preparation of Samples	Accuracy and Representativeness	(1) All aqueous and solid samples must be prepared (digested) prior to analysis. See SW-846 7470A and 7471B for details.	No	NA	NA	NA
Initial Calibration	Laboratory Analytical Accuracy	(1) Frequency: Daily prior to sample analysis. (2) Minimum calibration blank plus 5 calibration standards (multi-point); high level standard in calibration defines the upper end of the linear calibration range. (3) Linear regression with correlation coefficient $r \geq 0.995$.	No	NA	Perform instrument maintenance as necessary; re-optimize instrument; re-calibrate as required by SW-846 7470A and 7471B.	Suspend all analyses until initial calibration meets criteria.
Initial Calibration Verification (ICV)	Laboratory Analytical Accuracy	(1) Frequency: Immediately after each initial calibration. (2) Prepared using standard source different than used for initial calibration. (3) Concentration level near midpoint of curve. (4) Percent recovery must be between 90-110%.	No	NA	(1) Reanalyze ICV; if acceptable, no further action required. (2) If reanalysis is still outside of criteria, recalibrate and reanalyze ICV.	Suspend all analyses until ICV meets criteria.
Initial Calibration Blank (ICB)	Laboratory Analytical Sensitivity (instrument drift & contamination)	(1) Frequency: Immediately after ICV. (2) Prepared using same concentration of acids as calibration standards. (3) Mercury must be <RL.	No	NA	(1) Reanalyze ICB; if acceptable, no further action required. (2) If reanalysis is still outside of criteria, recalibrate and reanalyze ICV & ICB.	Suspend all analyses until ICB meets criteria.

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Table III B-1: Specific QC Requirements and Performance Standards for Mercury (SW-846 7470A & 7471B) Using WSC-CAM-III B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
Low-Level Calibration Verification (LLCV)	Laboratory Analytical Sensitivity (verify low-end of calibration range / verify RL)	(1) Frequency: Daily prior to sample analysis if initial calibration did not contain a low-level standard at the RL. If initial calibration includes the RL as the low-level standard in the initial calibration curve, then LLCV is not required. (2) Prepared using same source as initial calibration standards. (3) Concentration level must be at the level of the RL for mercury. (4) Percent recovery must be 70-130%.	No	NA	(1) Reanalyze LLCV; if acceptable, no further action required. (2) If reanalysis is still outside of criteria and concentrations of mercury are ≤10x RL in associated field samples, recalibrate and reanalyze LLCV and associated samples. (3) If concentrations of mercury are >10x RL in associated field samples, include explanation in laboratory narrative; no further action required.	Suspend all analyses until LLCV meets criteria unless the concentrations of mercury are >10x RL in associated field samples.
Continuing Calibration Verification (CCV)	Laboratory Analytical Accuracy	(1) Frequency: Every 10 samples and at the end of the analytical run. (2) Prepared using same source as initial calibration standards. (3) Concentration level near midpoint of curve. (4) Percent recovery must be 80-120%.	No	NA	(1) Reanalyze CCV; if acceptable, no further action required. (2) If reanalysis is still outside of criteria, recalibrate and reanalyze all associated samples since last compliant CCV – unless (3) applies. (3) If recovery is high (>120%) and all associated sample results are non-detected, no corrective action required.	If (3) applies, include explanation in laboratory narrative.

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Table III B-1: Specific QC Requirements and Performance Standards for Mercury (SW-846 7470A & 7471B) Using WSC-CAM-III B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
Continuing Calibration Blank (CCB)	Laboratory Analytical Sensitivity (instrument drift & contamination)	(1) Frequency: Every 10 samples following CCV and at the end of the analytical run. (2) Prepared using same concentration of acids as calibration standards. (3) Mercury must be <RL.	No	NA	(1) Reanalyze CCB; if acceptable, no further action required. (2) If reanalysis still outside of criteria, recalibrate and reanalyze all associated samples since last compliant CCB – unless (3) applies. (3) If concentration of mercury in CCB is >RL but all associated sample results are either non-detected or >10x concentration of mercury in CCB, no corrective action required.	If (3) applies, include explanation in laboratory narrative.
Method Blank (MB)	Laboratory Method Sensitivity (contamination evaluation)	(1) Frequency: One per digestion batch of ≤20 field samples. (2) Must be digested with the samples using the same preparation method as the samples. (3) Mercury must be <RL.	Yes	NA	(1) Reanalyze MB; if acceptable, no further action required. (2) If reanalysis is still outside of criteria, redigest and reanalyze MB and all associated field samples in batch – unless (3) applies. (3) If concentration of mercury in MB is >RL but all associated sample results are either non-detected or >10x concentration of mercury in MB, no corrective action required.	If (3) applies, include explanation in laboratory narrative.

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Table III B-1: Specific QC Requirements and Performance Standards for Mercury (SW-846 7470A & 7471B) Using WSC-CAM-III B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
Laboratory Control Sample (LCS)	Laboratory Analytical Accuracy	(1) Frequency: One per digestion batch of ≤20 field samples. (2) Must be matrix-matched by digesting with the samples using the same preparation method. CAM requires a solid Standard Reference Material (SRM) be prepared and analyzed with solid field samples as the "solid LCS." An SRM is a soil or sediment matrix that contains mercury at a known concentration and with 95% confidence limits. (3) Concentration level for aqueous LCS near midpoint of curve. (4) Percent recovery for mercury must be 80-120% for aqueous LCS and within vendor control limits (95% confidence limits) for solid LCS.	Yes	Aqueous LCS: Recovery <50%: mercury results in associated samples may be rejected.	(1) Reanalyze LCS; if acceptable, no further action required. (2) If reanalysis is still outside of criteria and LCSD is in-control for mercury, no corrective action required. (3) If LCS and LCSD are both outside of criteria, redigest and reanalyze LCS/LCSD and all associated field samples in batch.	Report recovery exceedances in laboratory narrative.
LCS Duplicate (LCSD)	Laboratory Analytical Accuracy & Precision	(1) Frequency: One per digestion batch of ≤20 field samples ONLY if not performing project-specific MD. (2) Must be matrix-matched by digesting with the samples using the same preparation method. CAM requires a solid SRM be prepared and analyzed with solid field samples as the "solid LCSD." An SRM is a soil or sediment matrix that contains mercury at a known concentration and with 95% confidence limits. (3) Concentration level must be same as LCS. Analyze immediately following LCS. (4) Percent recovery for mercury must be 80-120% for aqueous LCS and within	Yes ONLY if no MD	Same as above for LCS for recovery evaluation	(1) Reanalyze LCSD; if acceptable, no further action required. (2) If reanalysis is still outside of recovery criteria and LCS is in-control for mercury, no corrective action required. (3) If LCSD and LCS are both outside of recovery criteria, redigest and reanalyze LCS/LCSD and all associated field samples in batch.	Report recovery and RPD exceedances in laboratory narrative.

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Table III B-1: Specific QC Requirements and Performance Standards for Mercury (SW-846 7470A & 7471B) Using WSC-CAM-III B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
		vendor control limits (95% confidence limits) for solid LCS. (5) RPDs must be ≤20 for aqueous LCS/LCSD and ≤30 for solid LCS/LCSD.				
Matrix Spike (MS) Project-Specific	Method Accuracy in Sample Matrix	(1) Solid Samples (Soil/Sediment) Frequency: One per 20 field samples per matrix; designated by data user on COC or at project set-up. Aqueous Samples Frequency: One per digestion batch of ≤20 field samples per matrix strongly recommended (designated by data user on COC or at project set-up). (2) Concentration levels near midpoint of curve. (3) Percent recovery for mercury must be 75-125%.	Yes ONLY when requested by the data user	Recovery <30%: affects non-detects for mercury in all associated samples.	(1) Reanalyze MS; if acceptable, no further action required. (2) After reanalysis, if MS recovery is 30-74% or >125% and LCS was in-control, no corrective action is required. (3) If MS recovery is <30% and associated with non-detected results, redigest (homogenize sample well) and reanalyze sample/MS pair. Report results and narrate.	Report MS exceedances in laboratory narrative. If redigested due to recoveries <30%, report both sets of sample/MS data.
Matrix Duplicate (MD) Project-Specific	Method Precision in Sample Matrix	(1) Frequency: One per digestion batch of ≤20 field samples per matrix is strongly recommended (designated by data user on COC or at project set-up). (2) Prepare by digesting and analyzing an additional aliquot of the same field sample used for MS. (3) RPD for mercury must be ≤20 for aqueous and ≤35 for solids.	Yes ONLY when requested by the data user	NA	Narrate.	Report exceedances in laboratory narrative.

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Table III B-1: Specific QC Requirements and Performance Standards for Mercury (SW-846 7470A & 7471B) Using WSC-CAM-III B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
General Reporting Issues	NA	(1) Non-detected values must be reported with the sample-specific RL for mercury using all appropriate preparation/dilution factors. (2) The laboratory must only report values ≥ the sample-specific RL. (3) Sample concentrations that exceed the highest calibration standard must be diluted and reanalyzed to fall within the linear calibration range. (4) Results for soils/sediments must be reported on a dry-weight basis for comparison to MCP regulatory standards. (5) Results must be reported with 2 or more "significant figures" if ≥RL. If reporting values below the RL, report with 1 or more "significant figures".[2] (6) Refer to Appendix III B-1 for chain-of-custody requirements regarding preservation, cooler temperature, and holding times.	NA	NA	NA	(1) The performance of dilutions must be documented in the laboratory narrative or on the report form. Unless due to elevated concentrations of mercury, reasons for dilutions must be explained in the laboratory narrative. (2) If samples are not preserved properly or are not received with an acceptable cooler temperature, note the non-conformances in the laboratory narrative. (3) If samples are prepared and/or analyzed outside of the holding time, note the non-conformances in the laboratory narrative. (4) Narrate any additional method non-compliance or sample-specific anomaly.

[1] As per Appendix IV of MassDEP Policy #WSC-07-350, *MCP Representativeness Evaluations and Data Usability Assessments*, September 2007, if these results are observed, data users should consider nondetect results as unusable and detected results as estimated with a significant low bias.

[2] Reporting protocol for "significant figures" is a policy decision included for standardization and consistency for reporting of results and is not a definition of "significant" in the scientific or mathematical sense.

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ATTACHMENT B

Procedure for Recording Sample Information Electronically

1. After samples have been logged into the LIMS, create a Batch and Bench Sheet.

2. Export preparation sequence from LIMS to Network Directory Q:\Metals\Log Books\ Category (e.g. Metals Prep Aqueous) \File No., by highlighting the Batch No. in the Navigation Pane and clicking on EXPORT at the topof the screen and saving in the appropriate folder.

3 Access the File from the Tablet and enter Sample Data into the pertinent fields of the MS Excel spreadsheet. All information normally entered into a paper logbook will be entered here.

4. Go to the appropriate Bench Sheet, in the LIMS and click OPEN at the bottom of the screen. Double click the correct batch ID and the information is automatically entered into the LIMS.

5. Click SAVE.

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Division of Thielsch Engineering
Cranston, RI

SOP NO. 30_ 3005
Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by GFAA, FLAA or ICP Spectroscopy (EPA 200.7 PREP/SW846 3005A/ SW846 3020A)

APPROVED BY:

Operations Manager — Date 7/7/10

QA Officer — Date 6/30/10

Laboratory Director — Date 7/1/10

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Acid Digestion of Waters for Total Recoverable or Dissolved Metals by GFAA, FLAA or ICP Spectroscopy

1.0 SCOPE AND APPLICATION

1.1 Methods 200.7/3005A/3020A are acid digestion procedures used to prepare drinking, surface and ground water samples for analysis by flame atomic absorption spectroscopy (FLAA), furnace atomic absorption spectroscopy (GFAA) or by inductively coupled argon plasma spectroscopy (ICP). Samples prepared by Method 200.7/3005A may be analyzed by AAS or ICP for the following metals:

Aluminum	Calcium	Magnesium	Sodium
Antimony	Chromium	Manganese	Thallium
Arsenic	Cobalt	Molybdenum	Tin
Barium	Copper	Nickel	Titanium
Beryllium	Iron	Potassium	Vanadium
Boron	Lead	Selenium	Zinc
Cadmium	Lithium	Silver	

1.2 Samples prepared by Method 3005/3020A may be analyzed by GFAA for the following metals:

Arsenic	Chromium	Molybdenum	Thallium
Beryllium	Cobalt	Selenium	Vanadium
Cadmium	Lead	Silver	

1.3 When analyzing for total dissolved metals filter the sample, at the time of collection, prior to acidification with nitric acid. The detection limit is dependent on the determinative method used.

2.0 METHOD SUMMARY

2.1 Total recoverable metals: The entire sample is acidified at the time of collection with nitric acid. At the time of analysis, the sample is heated with acid and substantially reduced in volume. The digestate is filtered and diluted to volume, and is ready for analysis.

2.2 Dissolved metals: The sample is filtered through a 0.45 µm filter at the time of collection and the liquid phase is then acidified at the time of collection with nitric acid. Samples for dissolved metals do not need to be digested as long as the acid concentrations have been adjusted to the same concentrations as the standards.

3.0 HEALTH AND SAFETY

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3.1 Each employee has been trained and has acknowledged being trained in the safe use and handling of chemicals being used in the laboratory. This training has been performed according to the ESS Training SOP NO. 80_0016 and by the Chemical Hygiene Plan, SOP NO. 90_0001, in conjunction with the Safety orientation.

3.2 All sample and material handling should be done in a hood while using proper protective equipment to minimize exposure to liquid or vapor. Minimum personnel protective equipment includes the use of laboratory safety glasses, a lab coat or apron, and protective gloves.

3.3 The Material Safety Data Sheet (MSDS) for the concentrated chemicals used in the laboratory are kept on file and are available for all employees to review.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

4.1 For the determination of total recoverable elements in aqueous samples, samples are not filtered, but acidified with nitric acid to pH <2 (normally, 1 ml of acid per liter of sample is sufficient for most ambient and drinking water samples). Preservation is performed at the time of collection. Following acidification, the sample should be mixed, held for sixteen hours, and then verified to be pH <2 just prior to withdrawing an aliquot for processing or "direct analysis." If for some reason such as high alkalinity the sample pH is verified to be > 2, more acid must be added and the sample held for sixteen hours until verified to be pH < 2.

NOTE: When the nature of the sample is either unknown or is known to be hazardous, acidification should be done in a fume hood.

4.2 Hold time for aqueous preserved samples is 180 days from day of sampling. Samples prepared after this date are to be flagged as estimated values.

4.3 For dissolved metals analysis a non-preserved sample must be filtered through a 0.45 μm filter within 24 hours of collection and preserved to a pH of less than two.

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

5.1 The analyst should be cautioned that this digestion procedure might not be sufficiently vigorous to destroy some metal complexes. Precipitation will cause a lowering of the silver concentration and therefore an inaccurate analysis.

5.2 Primarily, the problems encountered in this step of the analysis are due to sample viscosity, sample homogeneity and sample concentration issues. Most are dealt with by dilution, but all decisions should be discussed with the project manager.

5.3 All samples are well mixed prior to sub-sampling to minimize sample homogeneity issues.

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6.0 EQUIPMENT/APPARATUS

6.1 **Volumetric Flasks,** various volumes

6.2 Color-coded **labels** or **tape**. (Yellow – Aqueous)

6.3 **Metals Digest Logbook**.

6.4 Finntip or Eppendorf **micropipette** calibrated according to SOP NO. 110.0005.

6.5 **Hot Block**, digester by Environmental Express

6.6 **Disposable digestion vessels**, by Environmental Express

6.7 **Disposable ribbed watchglass**, by Environmental Express

6.8 **Disposable screw cap covers**, by Environmental Express

6.9 **0.2 micron FilterMates**, by Environmental Express

7.0 REAGENTS AND STANDARDS

7.1 **Reagents**: All reagents used must be trace-metal grade and interference free. Reagents must be checked for contamination prior to use per SOPs 30.0003 and 80.0013 and must meet the criteria set forth in SOP 80.0013.

7.1.1 **De-ionized water (DI)**: DI water is prepared per SOP No 110.0003.

7.1.2 **Concentrated Nitric Acid**.

7.1.3 **1:1 Hydrochloric Acid**: In a 1 L volumetric flask, add 500 mL of DI water then carefully add 500 mL of acid and mix.

7.2 Standards:

7.2.1 **Primary Mixed Standard**: CM#1 purchased from VHG.

7.2.2 **Secondary Mixed Standards**: purchased from CPI. These standards contain all of the target analytes.

7.2.3 **Intermediate Secondary Mixed Standards**: Add 0.5 ml of Secondary Mixed Standard (see 7.2.2) to a 100 ml volumetric flask and dilute to the mark with ICP solution.

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7.2.4 **Blank Spike/Blank Spike Duplicate (BS/BSD)**: To 50 ml of reagent water added to a digestion vessel, add 0.25 ml of Secondary Mixed standards (Section 7.2.2). This undergoes the same preparation procedure as a typical sample.

7.2.5 **Blank Spike/Blank Spike Duplicate Low level (BS/BSD)**: To 50 ml of reagent water added to a digestion vessel, add 2 ml of Intermediate Secondary Mixed standard. This undergoes the same preparation procedure as a typical sample.

8.0 PROCEDURE

8.1 Obtain a batch number from the Element system for sample digestion. Ex: BG52701 where B = Batch, G = Month, 5 = Year, 27 = Date, 01 = batch number for that day.

8.2 **HOT BLOCK Procedure:** Turn Hot Blocks on. Prepare a disposable digestion vessel with 50 ml of DI water containing 1ml of HNO_3 and 5ml of 1:1 HCl and place in chamber (This solution will be used to measure chamber temperature).

8.3 Record the temperature of hot plate or Hot Block chamber in addition to all the pertinent sample information in the Metals Digestion Logbook. See Attachment A. Alternatively, record the information using an electronic device, such as an HP Tablet, as described in Attachment B.

8.4 Using tape, label digestion vessel with the ID# of their corresponding sample number, e.g., "0507001-01".

8.5 For each sample, shake thoroughly, trying not to aerate. Measure 50 ml (or 100 ml if needed) of sample directly into digestion vessel.

8.6 For metals that are to be analyzed, add 1 ml of concentrated HNO_3 and 5 ml of 1:1 HCl. The sample is covered with a ribbed watch glass or other suitable covers and heated on a Hot Block at 90-95 °C until the volume has been reduced by half. (Caution: Do not boil. Antimony is easily lost by volatilization from hydrochloric acid media.)

8.7 Volumize sample to a final volume of 50 ml (occasionally 25 ml, if needed) and if necessary, filter using a 0.2 micron FilterMate by attaching disposable plunger to filter and forcing filter to bottom of digestion vessel. The FilterMate will remain on bottom of vessel. Detach plunger and discard.

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8.8 The prep date is written on the cap, using a permanent marker. The sample extract is now ready for analysis.

9.0 CALCULATIONS (DATA INTERPRETATION)

9.1 Not Applicable.

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10.0 QUALITY ASSURANCE/QUALITY CONTROL

10.1 Method Blank (MB).

10.1.1 A method blank must be run with every batch not to exceed 20 samples.

10.1.2 The method blank is 50 ml of DI water, and undergoes the same preparation procedure as a typical sample.

10.1.3 The method blank acceptance criterion is less than ½ MRL for each metal to be reported.

10.2 Blank Spike/Blank Spike Duplicate (BS/BSD).

10.2.1 A BS/BSD must be run with every batch not to exceed 20 samples.

10.2.2 The limits of acceptance are found in the applicable instrument SOP along with corrective actions. All of the target analytes must be spiked.

10.3 Matrix Duplicate (MD) and Matrix Spike (MS) Samples.

10.3.1 A matrix duplicate and a matrix spike are run with every batch not to exceed 10 samples. All of the target analytes must be spiked.

10.3.1.1 The matrix duplicate is an additional aliquot of sample that is carried through the same preparation procedure as a typical sample.

10.3.1.2 The matrix spike is a sample that is measured out, spiked with 2 ml of Intermediary Secondary Mixed Standard for the MSLL (Section 7.2.3) or 0.25 ml of secondary mixed standard for the MS (7.2.2), and undergoes the same preparation as the original sample (where final volume is 50 ml; adjust these amounts if final volume is 25 ml)

10.4 Analysts are required to prepare four BS/BSDs to demonstrate precision and accuracy, thereby showing proficiency with this method. All of the target analytes must be spiked.

11.0 DATA VALIDATION

11.1 All unusual observations and method deviations will be noted in the narrative accompanying the data report presented to the client. See the applicable determinative SOP for appropriate corrective actions.

11.2 Refer to determinative method for validation data.

12.0 REFERENCES

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12.1 USEPA OSW SW-846 3rd edition, Update IV Methods 3005A/3520A: Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy and applicable sections in Chapter One.

12.2 NELAC Quality Systems Chapter 5, June 2003

12.3 DOD Quality Systems Manual, Final Version 4.2, October 2010.

12.4 Methods for the Determination of Metals in Environmental Samples Supplement I. EPA/600/R-04/111 May 1994.

13.0 POLLUTION PREVENTION and WASTE MANAGEMENT

13.1 ESS Laboratory's policies on pollution prevention and waste management are covered in SOP 90_0002, Hazardous Waste Contingency and Emergency Response Plan. All employees are trained in the requirements of the SOP.

14.0 DEFINITIONS

14.1 **Calibration Blank (CB)** -- A volume of reagent water fortified with the same matrix as the calibration standards, but without the analytes.

14.2 **Calibration Standard (CAL)** -- A solution prepared from the primary dilution standard solution or stock standard solutions. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

14.3 **Blank spike (BS)** -- An aliquot of reagent water or other blank matrices to which known quantities of the method analytes are added in the laboratory. The BS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.

14.4 **Matrix spike (MS)** -- An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The MS is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS corrected for background concentrations.

14.5 **Method Blank (MB)** -- An aliquot of reagent water or other blank matrices that are treated exactly as a sample including exposure to all glassware, equipment, and reagents that are used with other samples. The MB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

14.6 **Method Detection Limit (MDL)** -- The minimum concentration of an analyte that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero.

14.7 **Quality Control Sample (QCS)** -- A solution of method analytes of known concentrations that is used to fortify an aliquot of BS or sample matrix. The QCS is obtained from a source external

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14.7 **Quality Control Sample (QCS)** -- A solution of method analytes of known concentrations that is used to fortify an aliquot of BS or sample matrix. The QCS is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check laboratory performance with externally prepared test materials.

14.8 **Stock Standard Solution (SSS)** -- A concentrated solution containing one or more method analytes prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source.

15.0 PERSONNEL QUALIFICATIONS

15.0 Analysts who perform this analysis must have a working knowledge of quantitative and qualitative analysis, instrumental methods of analysis, chemical laboratory methods and equipment.

15.1 All analysts, before performing any analysis, participate in the ESS Laboratory training program (SOP80_0016). The training process consists of reading the Standard Operating Procedure, gaining instruction on the procedure from an experienced analyst, and performing the initial demonstration of capability.

16.0 TROUBLESHOOTING

Refer to the determinative method.

17.0 Data Management And Records

17.1 **Data Management** - ESS Laboratory has adopted the Promium Element LIMS system as part of its Data Management system. Sample information is entered into the LIMS and analyses are assigned to each sample. The LIMS allows EPA hold times and QC criteria to be assigned to each analysis. Standards can be entered and assigned to QC samples through the LIMS. Once analysis has been performed, data is imported using DataTool avoiding manual errors. In conjunction with Crystal Reports, the ELEMENT system allows for a wide variety of reporting formats.

17.2 **Records** – The specific retention periods required in the NELAC Standards, EPA-CFR and state and local statutes are followed or exceeded. At a minimum, data records are retained for five years from last use (10 years for drinking water and Mass DEP records). In cases of retention time uncertainty the record is kept until retention period is determined. Records are stored in specified-labeled locations and are easily retrievable. All raw data associated with testing is also retained including; computer printouts, chromatograms, review forms, and logbooks.

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18.0 ATTACHMENTS

18.1 Attachment A – Metals Prep Logbook.

18.2 Attachment B -- Procedure for recording sample information electronically.

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ATTACHMENT A

ESS LABORATORY

METALS PREP LOGBOOK

ANALYST:

DATE:

TIME:

Batch ID:

HNO_3 Reagent - AR# : _____

1:1 HCl Reagent- WR#: _____

1:1 HNO_3 Reagent- WR#: _____

H_2O_2 Reagent- AR#: _____

Hot Plate	Temp (°C)

Sample ID	matrix	pH	Initial wgt/vol	Final wgt/vol	QC ID/Lot #	QC wgt/vol	Method	Hot Plate Number	Comments

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CONTROL# 30.0014-0604A Page_____

MATRIX KEY: AQ = AQUEOUS, S = SOIL, O = OIL, F = FILTER, D = SLUDGE

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ATTACHMENT B

Procedure for Recording Sample Information Electronically

1. After samples have been logged into the LIMS, create a Batch and Bench Sheet as described in the attached instructions.

2. Export preparation sequence from LIMS to Network Directory Q:\Metals\Log Books\ Category (e.g. Metals Prep Aqueous) \File No., by highlighting the Batch No. in the Navigation Pane and clicking on EXPORT at the topof the screen and saving in the appropriate folder.

3 Access the File from the Tablet and enter Sample Data into the pertinent fields of the MS Excel spreadsheet. All information normally entered into a paper logbook will be entered here.

4. Go to the appropriate Bench Sheet, in the LIMS and click OPEN at the bottom of the screen. Double click the correct batch ID and the information is automatically entered into the LIMS.

5. Click SAVE.

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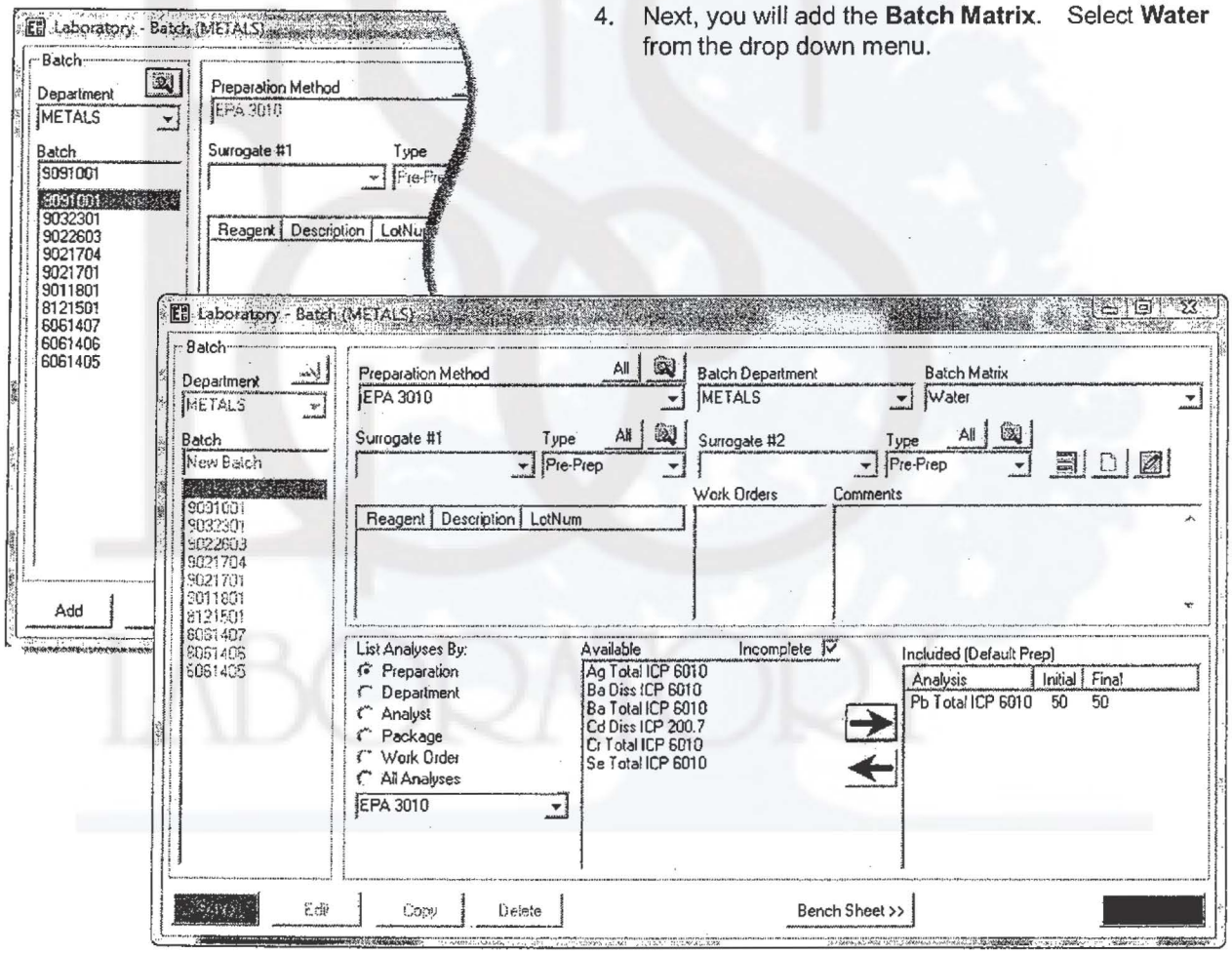
Step 6 Create Batches and Bench Sheets

Once the samples are logged in, samples are batched for prep and analysis. The Batch screen is like the header information from your current preparation logbook, and the bench sheet is the listing of the samples corresponding to that batch.

1. To **Batch** samples, go to the **Laboratory** menu and select **Batch**. This will open a new window.
2. Select **METALS** from the Department drop down and then click the **Add** button.
3. Now select the preparation method. Go to the drop down for **Preparation Method** and select **EPA 3010**, or you can click on the browse folder icon, select Extraction to sort the choices, and then double click on EPA 3010.
4. Next, you will add the **Batch Matrix**. Select **Water** from the drop down menu.



5. Finally, you will need to select the appropriate Analysis. Make sure the **Preparation** radio button is selected in the **List Analyses By**: section.
6. Select the **Incomplete check box** in the middle of the screen to provide a list of analyses of samples logged in that have not yet been batched.
7. Select **Pb Total ICP 6010** and double click, or use the right arrow.
8. Then select **Save**.

Take note of the Batch number highlighted in the Batch field. This will be your batch for this demonstration session

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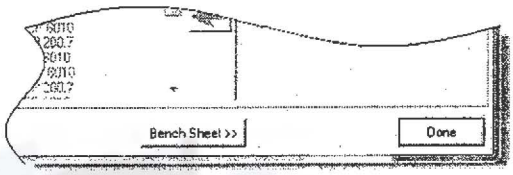
Step 6 Create Batches and Bench Sheets

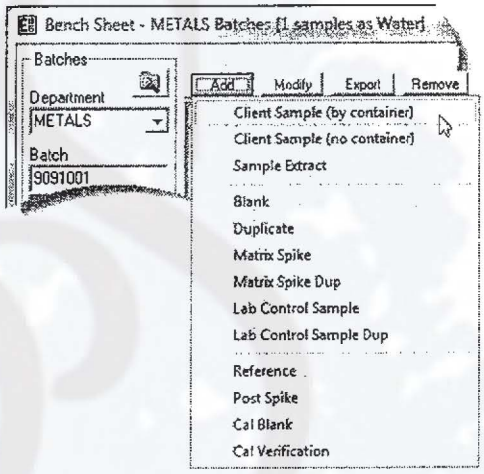
Now you will create an electronic Bench Sheet as part of the same workflow process.

1. Select the **Bench Sheet >>** button at the bottom of the screen. If a dialogue box appears asking you if you wish to Save Changes, select **Yes.**

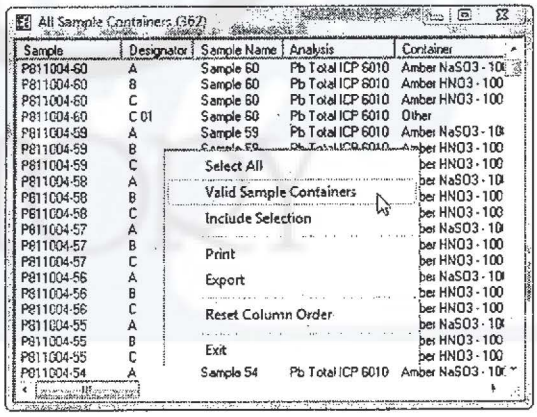


2. Select **METALS** (should already be highlighted) from the Department drop down and then select **Edit** at the bottom of the screen.

3. Next, click the **Add** button. Select **Client Sample by Container** and a new window will open.



4. To filter out containers not valid for this analysis, **right click** anywhere in this new window and select **Valid Sample Containers.** Double click on the highlighted sample. Close this window (click on X in top right corner). The Client Sample is added to the bench sheet and at the same time, QC samples are added. *Default QC samples are defined during the Analysis set up.*



WEB VIEW

TIP: *Element uses colored text to indicate conditions that require further action. Here, red text indicates items with expired hold times. Purple text identifies items with hold times due to expire in 24 hours. Green text identifies items within the proper hold times. If you see blue text, it has been edited or calculated.*

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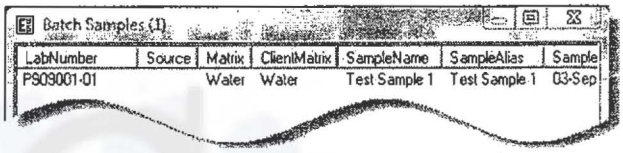
Step 6 Create Batches and Bench Sheets

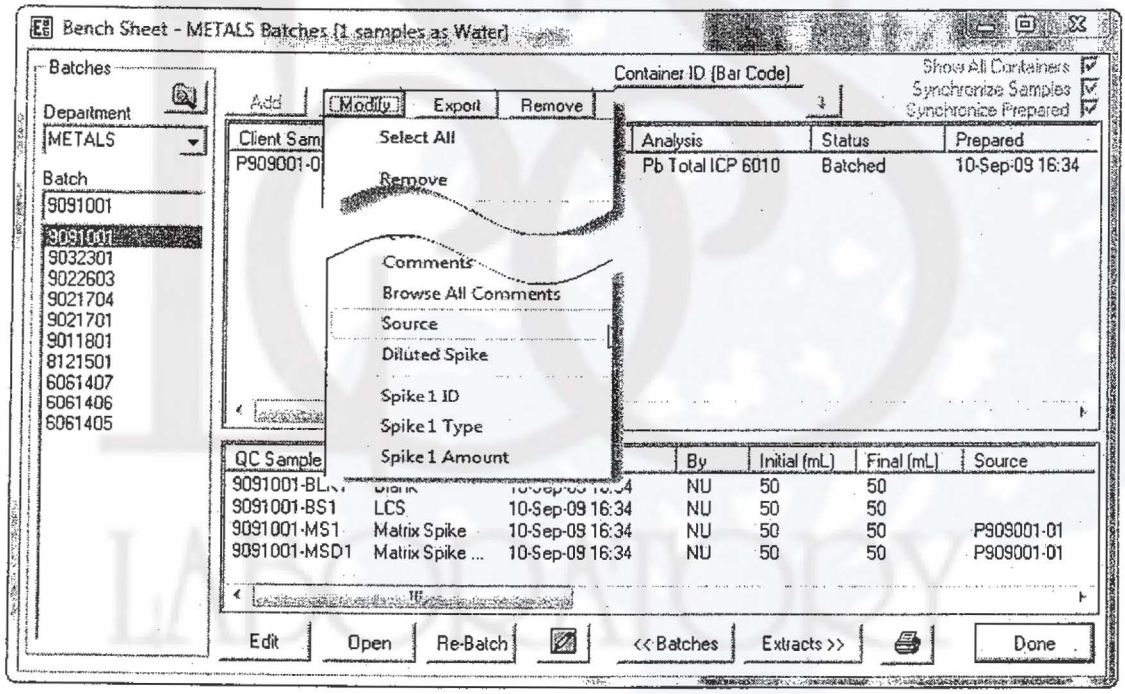
5. To associate Matrix Spikes and or duplicate QC Samples with a source sample, select the two **Matrix Spike** QC Samples from the lower portion of the screen. *Hold down the control key to select more than one at a time.*

6. Select the **Modify** button at the top of the screen (or right click in the highlighted portion).

7. Select **Source** from the pick list menu and a new window will open. Double click on the highlighted sample.



8. Now select **Matrix Spike, Matrix Spike duplicate,** and **LCS,** from the QC Sample field (hold the ctrl key down and select) and then select **Modify** again.



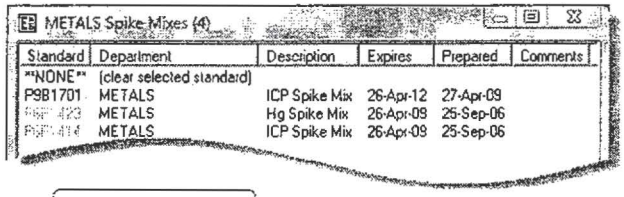
9. This time from the pick list drop down menu, select **Spike 1 ID** and a window will open.

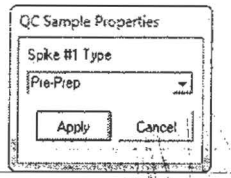
10. Select **P9B1701** ICP Spike Mix and double click.

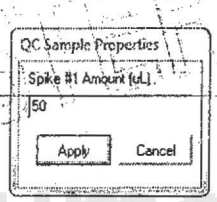
11. With the Matrix Spikes and LCS still selected, select **Modify** and then select **Spike 1 Type.** Select **Apply** for the dialogue box that opens.

12. Select **Modify** again and then select **Spike 1 Amount.** In the dialogue box that opens, enter a value of **50** and then **Apply.**

13. Select **Save** and then **Done.**







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ESS Laboratory
Division of Thielsch Engineering
Cranston, RI

SOP NO. 30_6010
INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROSCOPY
(SW 846 METHOD 6010B,C/ EPA METHOD 200.7/MCP-CAM IIIA)

APPROVED BY:

Operations Manager	Date 7/15/10
QA Manager	Date 7/15/10
Laboratory Director	Date 7/15/10

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INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROSCOPY (SW 846 METHOD 6010B,C/ EPA METHOD 200.7/MCP-CAM IIIA)

1.0 SCOPE AND APPLICATION

1.1 Inductively coupled argon plasma analysis (ICP) determines trace elements, including metals, in solution. The method is applicable to all of the elements listed in Table 3. All matrices, including ground water, aqueous samples, TCLP extracts, industrial and organic wastes, soils, sludge, sediments, and other solid wastes, require digestion prior to analysis.

1.2 Elements for which Method 6010/ 200.7 is applicable are listed in Table 3. Detection limits, sensitivity, and optimum ranges of the metals will vary with the matrices. The data shown in Table 3 provide typical reporting limits for clean aqueous samples.

2.0 METHOD SUMMARY

2.1 Prior to analysis, samples must be solubilized or digested using sample preparation method 3005A or 3020 (SOP 30_3005 addresses both these methods) for aqueous and method 3050B (SOP 30_3050B) for solids.

2.2 When analyzing for dissolved constituents, acid digestion is not necessary if the samples are filtered and acid preserved prior to analysis.

2.3 Methods 6010B and C describes the simultaneous multi-elemental determination of elements by ICP. The method measures element-emitted light by optical spectrometry. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element-specific atomic-line emission spectra are produced by a radio frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer, and a photosensitive device monitors the intensities of the lines. Background correction is required for trace element determination. Background must be measured adjacent to analyte lines on samples during analysis. The position selected for the background-intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interference and reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. The possibility of additional interferences named in Section 5.0 should also be recognized and appropriate corrections made. Tests for their presence are described in Step 7.2.2.5.

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3.0 HEALTH AND SAFETY

3.1 Each employee has been trained and has acknowledged being trained in the safe use and handling of chemicals being used in the laboratory. This training has been

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performed according to the ESS Training SOP 80_0016 and by the Chemical Hygiene Plan, SOP No. 90_0001, in conjunction with the Safety orientation.

3.2 All sample and material handling should be done in a hood while using proper protective equipment to minimize exposure to liquid or vapor. Minimum personnel protective equipment includes the use of laboratory safety glasses, a lab coat or apron, and protective gloves.

3.3 The MSDSs for the concentrated chemicals used in the laboratory are kept on file in a central location that is available for all employees to review.

3.4 The laboratory employee should review proper emergency response to spills or injury prior to attempting this procedure. Employees must know the location of spill kits, eyewashes, showers, and fire fighting equipment. Employees must also have knowledge of disaster evacuation routes.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

4.1 Prior to analysis, all aqueous samples are to be preserved with nitric acid (HNO_3) to a pH of less than two. If upon receipt the pH requires adjusting, nitric acid is added to the sample container until the pH has stabilized at pH<2, the sample is then held for 16 hours prior to digestion.

4.2 After digestion, samples are stored in specimen containers or Hot Block Tubes.

4.3 Hold time for aqueous preserved samples is 180 days from day of sampling for all metals except mercury. The hold time for samples to be analyzed for mercury is 28 days. Samples prepared and/or analyzed after this date are to be flagged as estimated values.

4.4 Prior to use, all glassware will be soaked in a 10% HNO_3 bath for at least 15 minutes and rinsed a minimum of three times with ICP solution (refer to SOP 30_0001). The HNO_3 bath is checked for contamination on a weekly basis and recorded in the batch log. When aqueous samples are digested by the Hot Block procedure plastic ware is used, so this step may be omitted.

4.5 All results for solid samples are to be corrected for a dry weight determination at 105°C.

4.6 For dissolved metals analysis, a non-preserved sample must be filtered through a 0.45 µm filter within 24 hours of collection and preserved to a pH of less than 2.

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

5.1 Spectral interferences are caused by: (1) overlap of a spectral line from another element at the analytical or background measurement wavelengths; (2) unresolved overlap of molecular band spectra; (3) background contribution from continuum or

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recombination phenomena; and (4) stray light from the line emission of high-concentration elements. Computer correcting the raw data after monitoring and measuring the interfering element can compensate for spectral overlap. Unresolved overlap requires selection of an alternative wavelength. Background contribution and stray light can usually be compensated for by a background correction adjacent to the analyte line.

5.1.1 Analysts must verify the absence of spectral interferences from an element in a sample for which there is no instrument detection channel. Laboratory standard wavelengths are listed in Table 3.

5.1.2 Element specific interference is expressed as analyte concentration equivalents (i.e., false analyte concentrations) arising from 100 mg/L of the interference element. For example, assume that Cd is to be determined (at 226.502 nm) in a sample containing approximately 10 mg/L of Fe. 100 mg/L of Fe would yield a false signal for Cd equivalent to approximately 0.007 mg/L. Therefore, the presence of 10 mg/L of Fe would result in a false signal for Cd equivalent to approximately 1 ppb. The interference correction factors should be determined annually. The possibility of interferences other than those determined does exist. The analyst should be aware of these interferences when conducting analyses.

5.1.3 Generally, interferences are discernable if they produced peaks, or background shifts, corresponding to 2% to 5% of the peaks generated by analyte concentrations.

5.2 Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. This SOP uses yttrium as an internal standard to minimize this effect. Differences in solution volatility can also cause inaccuracies when organic solvents are involved. If physical interferences are present, they must be reduced by diluting the sample. A problem that can occur with high dissolved solids is salt build-up at the top of the nebulizer, which affects aerosol flow rate and causes instrumental drift. Changing the nebulizer and removing salt build-up at the tip of the torch sample injector can be used as a measure to control salt build-up. Control of the argon flow rate does improve instrument performance.

5.3 Chemical interferences include molecular compound formation, ionization effects, and solute vaporization effects. Normally, these effects are not significant with the ICP technique. If observed, they can be minimized by careful selection of operating conditions (incident power, observation position, and so forth), by buffering of the sample, by matrix matching, and by standard addition procedures. Chemical interferences are highly dependent on matrix type and the specific analyte element.

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6.0 EQUIPMENT/APPARATUS

6.1 **Inductively coupled argon plasma emission spectrometer with background correction.**

6.1.1 Perkin Elmer 4300DV Serial #077N1032302, using an AS-91 Autosampler and a Polyscience Chiller, Serial # G54802.

6.1.2 Perkin Elmer 3100XL Serial #069N8031701, using an AS-91 Autosampler and a Neslab Chiller, Serial # CFT-33.

6.2 **Gases**:

6.2.1 Liquid **Argon** gas supply.

6.2.2 Liquid **Nitrogen** gas supply.

6.3 **Auto-sampler**, AS-90.

6.4 **Class A volumetric flasks**, 50 ml, 100 ml and 250 ml

6.5 **Variable transfer pipettes**, 1.0 to 5.0 ml and 0.1 to 1.0 ml calibrated according to SOP 110.0005. Also, 0.01 ml to 0.1 ml micropipettes

6.6 A **temperature adjustable hot plate or hot block,** capable of maintaining a temperature of 95°C.

6.7 **Data system**:

6.7.1 **Computers:** The Metals laboratory has two ICP-AES systems analyzing per Method 200.7/6010B. One system is a DELL OptiPlex 170L computer with a Windows XP Pro/SP2 operating system and the other is a HP 5100 computer, again with XP Pro/SP2. All computer systems are networked to a Windows 2000 server, which is the destination of all files. A differential back-up is performed nightly and a full back is performed each weekend using Veritas Backup Exec to tapes. As the systems acquires and stores data onto the server, the server becomes full. The data is downloaded and archived onto CDs.

6.7.2 **Software:** ICP WINLAB32 with revision (3.1.0).

7.0 REAGENTS AND STANDARDS

7.1 **Reagents**: reagent grade or better chemicals are used in all tests. If the purity of a reagent is in question, it is analyzed for contamination.

7.1.1 **Hydrochloric acid (conc.),** HCl: Trace Grade.

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7.1.2 **Hydrochloric acid (1:1)**, HCl: Add 500 ml concentrated HCl to 400 ml water and dilute to 1 liter in an appropriate beaker. Never add water to acid.

7.1.3 **Nitric acid (conc.)**, HNO_3: Trace Grade.

7.1.4 **Nitric acid (1:1)**, HNO3: Add 500 ml concentrated HNO_3 to 400 ml water and dilute to 1 liter in an appropriate beaker. Never add water to acid.

7.1.5 **De-Ionized Water** (DI): The deionized water is prepared using the equipment described in SOP 110.0003 The conductivity must also be checked and recorded according to SOP 110.0003. The lab supervisor needs to be notified if the conductivity is > 1 µmhos/cm. At this point, the supervisor will arrange for the cartridges to be replaced, if deemed necessary.

7.1.6 **ICP solution**: Any type of diluting (primary standards or samples) is done using ICP solution. ICP Solution is prepared by adding 400 ml of nitric acid and 600ml of hydrochloric acid to 5.0 liters of DI water in a 20 liter carboy and bringing up to 20 liters with DI water.

7.1.7 **Ghost Wipe**: Environmental Express Part No. 4210.

7.2 **Standards**:

7.2.1 **Primary Stock Solutions:** Solutions are stored at room temperature and not used beyond the manufacturer's expiration date. See manufactures certificate of analysis for concentrations.

Standard	Vendor	Elements
Mixed Standard 1	VHG	Ag, Al, As, B, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Mo, Na, Ni, Pb, Sb, Se, Sn, Ti, Tl, V, Zn
ICCV	CPI	Ag, Al, As, B, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Mo, Na, Ni, Pb, Sb, Se, Sn, Ti, Tl, V, Zn

7.2.2 **Working standard solutions** are used to calibrate the instruments. They are prepared daily. Initial analyte concentrations are obtained from the bottle label or the vendor's certificate of analysis. Calibration concentrations may be viewed and/or adjusted in the calibration page of the method editor window (refer to ICP Winlab Software Guide).

7.2.2.1 **Standard 1**: Add 0.2 ml of Mixed Standard 1 (see 7.2.1) to a 200 ml volumetric flask and dilute to the mark with ICP solution.

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7.2.2.2 **Standard 2**: Add 1.0 ml of Mixed Standard 1 (see 7.2.1) to a 200 ml volumetric flask and dilute to the mark with ICP solution. This standard is also used as the **CCV** (continuing calibration verification).

7.2.2.3 **Standard 3**: Add 1.0 ml of Mixed Standard 1 (see 7.2.1) to a 100 ml volumetric flask and dilute to the mark with ICP solution.

7.2.2.4 **Standard 4**: Add 0.2 ml of Mixed Standard 1 (see 7.2.1) to 10 ml volumetric flask and dilute to the mark with ICP solution.

7.2.2.5 **Standard 5**: Add 0.5 ml of Mixed Standard 1 (see 7.2.1) to 10 ml volumetric flask and dilute to the mark with ICP solution.

ICP Standard concentration (mg/L)

Elements	Std 1	Std 2	Std 3	Std 4	Std 5
Be	0.01	0.05	0.10	0.20	0.50
Ag, Cd	0.05	0.25	0.50	1.00	2.50
As, B, Ba, Co, Cr, Cu, Mn, Mo, Ni, Pb, Sb, Sn, Ti, Tl, V, Zn	0.10	0.50	1.00	2.00	5.00
Se	0.20	1.00	2.00	4.00	10.00
Al, Fe	0.50	2.50	5.00	10.00	25.00
Ca, Mg	1.0	5.0	10.0	20.0	50.0
K, Na	5.0	25.0	50.0	100.0	250.0

NOTE: Standards 4 and 5 are used primarily for Ba, Pb, Zn but may also be used for other metals as long as all quality control criteria are met for the element.)

7.2.2.6 The **instrument check standard** (SCV1: initial calibration verification) is used to verify the instrument calibration. It is prepared by adding 0.5 ml of ICCV)see 7.2.1) to a 100 ml volumetric flask and diluting to the mark with ICP solution.

7.2.2.7 The **interference check solutions** contain known concentrations of interfering elements, and are run at the beginning of each analytical run in order to test correction factors.

7.2.2.7.1 **IFA** is prepared by adding 12.5 ml of Inorganic Venture's CLP-ICS-A to a 250 ml volumetric flask and diluting to the mark with ICP solution.

7.2.2.7.2 **IFB** is prepared by adding 12.5 ml of Inorganic Venture's CLPP-ICS-A and 1.25 ml of CLPP-ICS-B in a 250 ml volumetric flask and diluting to the mark with ICP solution.

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7.2.2.8 Three **CRL Standards** (at the lower limit of quantitation) are used to determine the detection limits below the lowest point of the calibration curve. Three solutions are prepared to cover the range of reporting limits.

7.2.2.8.1 **CRL1** is prepared by adding 25 ml of Standard 1 (see 7.2.2.1) to a 50 ml centrifuge tube and diluting to the 50 ml mark with ICP solution.

7.2.2.8.2 **CRL2** is prepared by adding 1 ml of Standard 3 (see 7.2.2.3 to a 50 ml centrifuge tube and diluting to the 50 ml mark with ICP solution.

7.2.2.8.3 **CRL3** is prepared by adding 0.5 ml of Standard 3 (see 7.2.2.3 to a 50 ml centrifuge tube and diluting to the 50 ml mark with ICP solution.

7.2.2.9 Low Level CCV (MCP & SW846-6010C)

7.2.2.9.1 Identical to CRL Standards (see § 7.2.2.8)

7.3 **Two types of blanks** are required for the analysis. The calibration blank is used in establishing the analytical curve, and as an initial and periodic check for contamination throughout the analytical run. The method blank ID is created through the Element LIMS and is linked to the Batch ID. For example, BG52701-BLK1 where B = Batch, G = Month, 5 = Year, 27 = Date, 01 = Batch Number.

7.3.1 The **calibration blank** (ICB1, CCB) is prepared using ICP solution. (See 7.1.6).

7.3.2 The **method blank** must contain all the reagents and in the same volumes as used in the processing of the samples. The method blank must be carried through the complete procedure and contain the same acid concentration in the final solution as the sample solution used for analysis.

7.4 **Internal standard** is added to all standards and samples automatically, by the instrument. A 5 ppm yttrium standard is used for the 3100XL and 4300DV . It is prepared by adding 5 ml of a 1000 ppm standard (purchased from Perkin Elmer – Cat# PE#N9300167) to a 1000 ml volumetric flask and diluting to the mark with ICP solution. Expiration of 5 ppm Yttrium standard is determined by the manufacturer and is recorded on the container label. The element intensity is used by the instrument as an internal standard to ratio the analyte intensity signals for both calibration and quantitation.

7.5 The **Manganese X, Y optimization standard** (1.0 ppm) is used to fine tune the X, Y adjustment of the torch. Prepared by adding 0.25 ml of 1000 ppm Manganese

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the mark with ICP solution. Expiration of 1 ppm Manganese standard is determined by the manufacturer and is recorded on the container label.

8.0 PROCEDURE

8.1 Preliminary treatment of most matrices is necessary because of the complexity and variability of sample matrices. Water samples that have been pre-filtered and acidified do not need acid digestion as long as the samples and standards are matrix matched and the dissolved solids are < 0.2% w/v. Solubilization and digestion procedures are presented in Sample Preparation Method SOP 30_3005A and 30_3050B. ***NOTE: It is ESS Laboratory's policy to digest all dissolved samples.***

8.2 Set up the instrument with proper operating parameters. The instrument must be allowed to become thermally stable before beginning (usually requiring at least 15 minutes of operation prior to calibration).

8.2.1 Operating Conditions: The analyst should follow the instructions provided by the instrument manufacturer. For operation with organic solvents, use of the auxiliary argon inlet is recommended, as is solvent-resistant tubing, increased plasma (coolant) argon flow, decreased nebulizer flow, and increased RF power to obtain stable operation and precise measurements. Sensitivity, instrumental detection limit, precision, linear dynamic range, and interference effects must be established for each analyte line on that particular instrument. All measurements must be within the instrument linear range where spectral interference correction factors are valid. The analyst must (1) verify that the instrument configuration and operating conditions satisfy the analytical requirements and (2) maintain quality control data confirming instrument performance and analytical results.

8.3 Optimize and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the typical mixed calibration standard solutions described in Step 7.2.2 and the manganese optimization standard described in Step 7.5.

8.3.1 Double click **Winlab 32** Icon. Wait for the status check to be completed for the instruments and accessories.

8.3.2 Select Method.

8.3.3 Click on **Plasma** Icon on tool bar. Turn the virtual switch to the **on** position or press **F9** to automatically ignite the plasma.

8.3.4 After the plasma has ignited, allow the instrument to stabilize for 30 minutes before calibration.

8.3.5 Place centrifuge tube containing 1.0 ppm Mn solution on the auto-sampler tray. Press F10 and select appropriate location or click on **Analysis** column above

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the tool bar. Choose **auto-sampler Go To location** and enter the appropriate auto-sampler location to move the arm to the Mn solution. Click on **tools** and choose **spectrometer control**. Click on **AlignView**. The optimization should be reported in the Mn Profile Logbook (See Attachment A).

8.3.6 Flush the system with the calibration blank (Step 7.3.1) between each standard or as the manufacturer recommends. Press F10 and select **Wash**. (Use the average intensity of multiple exposures for both standardization and sample analysis to reduce random error.) The calibration curve should consist of a blank and a minimum of three standards.

8.3.6.1 Click on **File**. Choose **New, Sample Info File**. Enter batch ID number, which is the date, followed by x for the 3100 instrument only, and then a through z depending on the file number for the day. (i.e. 092798xa – date, 3100 instrument, first file.)

8.3.6.2 Click on **Default setup for 3100XL or ESS setup of 4300DV** Icon. The starting location is always 9 (1-8 reserved for standard and QC). For **sample number range**, adjust the end indicator to equal the number of samples being analyzed. Enter the sample IDs in the **Sample ID** column.

8.3.6.3 For duplicates and spikes, click on the **Matrix Check Samples** box to the right of the duplicate or matrix spike ID.

8.3.6.3.1 For duplicates, select option **Duplicate** and make sure that the reference sample number corresponds to the original sample. Click **OK**.

8.3.6.3.2 DELETED

8.3.6.3.3 DELETED

8.3.6.3.4 DELETED

8.3.6.4 There are currently two methods being used on the ICP, **EVERYTHINGX** and **EVERYTHING-DV**. For both methods, click on **Analyze QC's before**. In the cell at the top of the column, enter 1,

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4,6-9,2,3,5,6 or select 00Daily Cal from open Sample Info File to calibrate before running samples.

8.3.6.5 For both methods, arrow down 10 cells and enter **after 5,6**. Repeat this for every ten cells. This will ensure that the CCV and CCB will run every 10 samples.

8.3.6.6 At the end of the run, enter **5,6,2,3**. This will finish off all QC that is required to run at the end of an analysis. These QC numbers are all defined in the method. QC will run before the wash step.

8.3.6.7 Click on **File, Save As, and Sample Info File**; enter same file # as batch ID.

8.3.6.8 Click on **AUTO** Icon. On the setup screen, click on **Method** Cell to choose the appropriate method.

8.3.6.8.1 Make certain that the **SIF ID** is correct in the SIF cell.

8.3.6.8.2 Open **Results Data Set Name** under Q:\Metals\Results ICP#\Results and assign the appropriate ID to the data set. The ID is the Batch ID with 'ad' added to the end (xad for 3100XL)

8.3.6.8.3 Make sure that the **Save Data** and **Print Log** option boxes are selected.

8.3.6.8.4 Click on **Analyze tab**. Click on **Print List** at bottom of analyze window and print this out. Click **Analyze All** to start the run.

8.4 Before beginning the sample run, check that the %RSD for each element in each standard is <5%, and then reanalyze the mixed calibration standard 2 as if it were a sample. Concentration values obtained should not deviate from the actual values by more than 10% for 6010B or 5% for 200.7. If they do, follow the recommendations of the instrument manufacturer to correct for this condition and then recalibrate.

8.5 Analyze the instrument check standard (see 7.2.2.6) and the calibration blank (see 7.3.1). Analyze CRL – 1, 2, 3 and Standard 1 (low level quantitation standard), then the IFB and IFA solutions (see 7.2.2.7.1 and 7.2.2.5.2) immediately before the first sample analysis. Analyze the continuing calibration standard (CCV, see 7.2.2.2) after every ten samples and at the end of the run.

8.6 Below is a typical analytical sequence for a batch of twenty samples:

- Standard Blank (S0)

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- Calibration Standard(s)
- Re-analyzed Calibration Standard; 2 ± 10%, RPD ≤5% (± 5% for 200.7)
- Instrument Check Standard (SCV1); ± 10%, RPD ≤5%
- Calibration Blank (ICB) <MDL
- CRL1 ±30% (DoD QSM 4.2 ±20%)
- CRL2 ±30% (DoD QSM 4.2 ±20%)
- CRL3 ±30% (DoD QSM 4.2 ±20%)
- Standard 1 ±30% (DoD QSM 4.2 ±20%)
- Interference Check Sample (IFA) ± MRL
- Interference Check Sample (IFB) ± 20%
- Method blank- BLK
- Blank spike- BS
- Blank spike duplicate- BSD
- Sample 1
- Matrix duplicate
- Matrix spike
- Serial dilution
- Post digestion spike
- Samples 2-3
- CCV
- CCB
- Samples 4-11
- Matrix duplicate
- Matrix spike
- CCV
- CCB
- Serial dilution
- Post digestion spike
- Samples 12-19
- CCV
- CCB
- CRL1 ±30% (DoD QSM 4.2 ±20%)
- CRL2 ±30% (DoD QSM 4.2 ±20%)
- CRL3 ±30% (DoD QSM 4.2 ±20%)
- Standard 1 ±30% (DoD QSM 4.2 ±20%)
- Sample 20
- CCV
- CCB
- Interference Check Sample (IFA) ± MRL
- Interference Check Sample (IFB) ± 20%

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8.7 All sample information is recorded in the run logbook (see Attachment B) and typed into the sample info file (SIF) on the ICP. The run log is used to load the auto-sampler tray. The sample information printout is compared to the run log for accuracy.

9.0 CALCULATIONS - Note: The instrument printout is in mg/L.

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9.1 All results should be reported with up to three significant figures.

9.2 The following calculation for **aqueous samples** will provide results in mg/L:

$$\text{Final conc.} = \frac{\text{mg/l (raw)} \times \text{dilution factor} \times \text{final volume}}{\text{Initial volume}}$$

9.3 The following calculation for **solid/soil samples** will provide results in mg/kg dry weight:

$$\text{Final conc.} = \frac{\text{mg/l (raw)} \times \text{dilution factor} \times \text{final volume (ml)}}{\text{Initial weight (g)/ (\%solids/100)}}$$

9.4 When reporting results, include date analyzed and initials of analyst on summary sheets and actual runs.

9.5 Hardness. The preferred method for determining hardness is to compute it from the results of separate determinations of calcium and magnesium (SM 2340B):

Hardness, mg equivalent $CaCO_3$ /L = 2.497 [Ca, mg/L] + 4.118 [Mg, mg/L]

9.6 The following calculation for **wipe samples** will provide results in µg/Sample:

Final conc. = mg/l (raw) x dilution factor x final volume (ml)

10.0 QUALITY ASSURANCE/QUALITY CONTROL

10.1 All quality control data should be maintained and available for easy reference or inspection. A summary of general method quality objectives may be found in Table 4.

10.2 The upper limit of the Linear Dynamic Range (LDR) must be established for each wavelength utilized. It must be determined from a linear calibration prepared in the normal manner using the established analytical operating procedure for the instrument. The Linear Dynamic Range should be determined by analyzing a minimum of three consecutively higher standard concentrations of the analyte until the observed analyte concentration is no more than ± 10% outside the stated concentration of the standard. Determined LDRs must be documented and kept on file. The analysis from the resulting data establishes the LDR, which may be used for the analysis of samples. Determined sample analyte concentrations that are greater than 90% of the determined upper LDR limit must be diluted and reanalyzed. The LDRs should be verified semi-annually or whenever, in the judgment of the analyst, a change in analytical performance caused by either a change in instrument hardware or operating conditions would dictate they be re-determined. Record is filed in Directory Q:\Quality\QA\LDR.

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10.3 Instrument detection limits (IDL) are calculated by running the calibration blank seven consecutive times. Calculate the standard deviation and multiply by 3 (or 3.14) to determine the IDL. IDL shall be ≤LOD. The IDL must be determined after initial set-up and after any significant change. Record is filed in Directory Q:\Quality\QA\IDL.

10.4 Calibration for the ICP consists of a blank and a minimum of three standards. The correlation coefficient (R) for each element calibration curve must be ≥ 0.995 (≥ 0.998 for 6010C and MCP).

10.5 Initial and Periodic Method QC Demonstrations: The procedures specified in Section 10.3.1 through 10.3.2 must be conducted as an initial demonstration of laboratory capability, prior to the analysis of any samples. Subsequent to this initial demonstration, additional evaluations of this nature should be conducted on a periodic basis, in response to changes in instrumentation or operations, and/or in response to confirmed or suspected systems, method, or operational problems.

10.5.1 Accuracy and Precision: To demonstrate initial laboratory capability, analyze a minimum of four initial calibration verification standards. The mean concentration must be within ± 10 % of the stated values. (± 5% for 200.7)

10.5.2 Method Detection Limits (MDL) for Individual Analytes: MDLs must be determined for each analyte/matrix/instrument combination at initial set-up and thereafter annually. See SOP 110_0013 for specific instruction on MDL determination.

10.6 Dilute and reanalyze samples that are more concentrated than the linear range study or use an alternate, less sensitive line for which quality control data is already established.

10.7 It is recommended that whenever a new or unusual sample matrix is encountered, or per preparatory batch at a minimum, a series of tests is performed prior to reporting concentration data for analyte elements. These tests, as outlined in Steps 10.7.1 and 10.7.2 will ensure the analyst that neither positive nor negative interferences are operating on any of the analyte elements to distort the accuracy of the reported values.

10.7.1 Serial dilution **(required by DOD QSM v.4.2; conditionally required by MCP – see Attachment D)**: If the analyte concentration is sufficiently high (50x the instrumental detection limit after dilution), an analysis of a 1+4 (5x) dilution should agree within ± 10% of the original determination. If not, a chemical or physical interference effect should be suspected.

10.7.1.1 Prepare the serial dilution by diluting the sample with ICP solution. If the sample was diluted to bring the result within the linear dynamic range, then the serial dilution must be performed on that dilution (Ex. Sample result based on 5x dilution, then serial dilution must be 25x for the applicable metals.)

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10.7.2 Post digestion spike addition **(not required by MCP)**: An analyte spike added to a portion of a prepared sample, or its dilution, should be recovered to within 85% to 115% (75-125% for method 6010B) of the known value. The spike addition should produce a minimum level of 20 times and a maximum of 100 times the instrumental detection limit. If the spike is not recovered within the specified limits, a matrix effect should be suspected.

10.7.2.1 Measure out 5 ml of prepared sample into a centrifuge tube. Add 0.05 ml of mixed standard 1 (see 7.2.1), then bring to 10 ml with sample. Mix and set up to run after the serial dilution. The post digestion spike is performed on that dilution that the sample result was obtained.

10.8 Check the instrument standardization by analyzing check standards as follows:

10.8.1 Verify calibration every 10 samples and at the end of the analytical run, using a calibration blank (Step 7.3.1) and a continuing calibration check standard (Step 7.2.2.2).

10.8.1.1 The results of the check standard (ICV and CCV) should agree within 10% of the expected value and %RPD ≤5%. See section 11.0 for corrective action for out of criteria results.

10.8.1.2 Method 6010C requires that a low level ICV and low level CCV be run, meeting an accuracy criteria of 30% **(20% per DoD QSM 4.2)** .

10.8.1.3 The results of the calibration blank should be no greater than the MRL **(≤ LOD in the case of Method 6010C or the DOD QSM)**. See section 11.0 for corrective action for out of criteria results.

10.8.2 Verify the interelement and background correction factors at the beginning and end of each analytical run (***the end interelement check is only required for MA MCP samples by method 6010B***). Do this by analyzing the interference check solution (Step 7.2.2.5). Results should be within ±20% for analytes found in the IFB and ± MRL, or **< the LOD if DoD QSM criteria need to be met**, for analytes not found in the IFA (verified trace impurity from one of the spiked analytes). If outside of limits, stop analysis and recheck background corrector points and/or interelement correction factors. Re-analysis interelement check solutions. Samples may not be analyzed without a valid interference check.

10.8.3 Replicate samples are to be analyzed at a frequency of 10% or per analytical batch, whichever is more frequent.

10.8.3.1 The relative percent difference between replicate determinations is to be calculated as follows:

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$$RPD = \frac{D_1 - D_2 \times 100}{(D_1 + D_2)/2}$$

Where:

RPD = relative percent difference
D_1 = first sample value
D_2 = second sample value (replicate)

10.8.3.2 A control limit of ±20% RPD shall be used for aqueous samples that are ≥5x the MRL and ±MRL for when <5x MRL. A control limit of ±35% RPD shall be used for soil/sediment samples that are ≥5x the MRL and ±2xMRL for when <5x MRL. ***Refer to Attachment D for DOD requirements and Attachment E for 6010C/MCP requirements.*** See section 11.0 for corrective action for out of criteria results.

10.8.4 Spiked samples are to be analyzed at a frequency of 10% or per analytical batch, whichever is more frequent.

10.8.4.1 The spiked sample recovery is calculated as follows:

$$\% \text{ Recovery} = \frac{SSR - SR}{SA} \times 100$$

Where:

SSR = Spiked Sample Result
SR = Sample Result
SA = Spike Added to Sample

10.8.4.2 A control limit of 25% shall be used for all sample matrices. See Table 2 for DoD control limits, sporadic marginal exceedance limits are same as for blank spike (section 10.10.2) See section 11.0 for corrective action for out of criteria results.

10.9 Employ a minimum of one method blank per sample batch to determine if contamination is occurring during sample preparation. The method blank must contain all the reagents and in the same volumes as used in the processing of the samples. It must be carried through the complete procedure as the sample solution used for analysis.

10.9.1.1 The value for the method blank should be less than the MRL **(≤ ½ the MRL in the case of DOD)**. See section 11.0 for corrective action for out of criteria results.

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10.10 At least one BS/BSD must be analyzed for each batch of samples. These should be prepared and analyzed in the same manner as the samples (see SOP 30_3005 and 30_3050B.

10.10.1 The BS/BSD recovery range must be within the range of 80-120% (85-115% for 200.7), ***see Table 2 for DoD blank spike recovery acceptance criteria.*** The RPD must be ≤20% for aqueous and ≤ 30% for soils, ***DoD %RPD <20% for all matrices.***

10.10.2 If the BS or BSD falls outside of this range, see section 11.0 for corrective action for out of criteria results.

10.11 Method Reporting Limit Standards (CRIs) are run at the beginning of every analytical run. Results should agree to ± 30% (**± 20% if DoD QSM Criteria are to be met**). If not, review blank data for contamination.

10.12 BS/BSDs are plotted in Element to determine if in-house limits are within default limits.

11.0 DATA VALIDATION

11.1 Data validation will be accomplished by reviewing all of the quality control parameters and assuring that they are within recommended ranges and recording any deviations in the ICP Tray Sequence Logbooks (Attachments B-1 and B-2). The only exceptions made to ranges would be the following:

11.1.1 For duplicates, ±20% RPD shall be used for aqueous samples that are ≥5x the MRL and ±MRL for when <5x MRL. A control limit of ±35% RPD shall be used for soil/sediment samples that are ≥5x the MRL and ±2xMRL for when <5x MRL. ***When analyzing DoD samples %RPD ≤ 20%.*** However, there are cases where duplicates may not work. If this is the case, inform client in narrative concerning sample non-homogeneity.

11.1.2 For matrix spikes, the % Recovery should be ±25%. If the matrix spike is >30%, check the LCS. If the LCS is within limits, matrix interferences are present and must be noted in the narrative. ***See Table 2 for DoD MS control limits.*** If the matrix spike is <30% and non-detected results were found re-digest sample and MS to confirm matrix interference(for DoD, work perform Method of Standard additions, see Addendum 1).

11.1.3 PDS and Serial dilution (section 10.7) are used to test the presence of matrix affect when the matrix spike or duplicate is outside criteria. When the PDS or SD is used, their failure is to be reported in the projective narrative as a confirmation of matrix affect.

11.1.4 For the BS/BSD, the % Recovery must be within 80-120% (85-115% for 200.7). ***See Table 2 for DoD MS control limits.*** If the BS or BSD is outside

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criterion, then re-prep and re-analyze samples with the following exception: ***For high BS/BSDs, those samples that are non-detects may be reported.***

11.1.5 Analytical batches with Method blanks above the MRL **(≤ ½ the MRL in the case of DOD)** (will be re-prepped and re-analyzed with the following exceptions:

11.1.5.1 Samples that are that are at least twenty times higher than the method blank may be reported.

11.1.5.2 When the method blank is less than 5% of the regulatory limit associated with the analyte the method blank would be acceptable.

11.1.5.3 If the analyte is found in the method blank above the MRL but is not in any of the associated samples, no corrective action is needed.

11.1.5.4 Any results that are reported with method blank contamination must be B-flagged.

11.1.6 All unusual observations and method deviations will be noted in the narrative accompanying the data report presented to the client.

11.2 All unusual observations and method deviations will be noted on the Metals Data Table (See Attachment C) and will be included in the project narrative accompanying the data report presented to the client.

12.0 REFERENCES

12.1 SW846 Methods 6010B and C, Inductively Coupled Plasma – Atomic Emission Spectrometry; third edition, Update III and SW846 On-line, respectively.

12.2 ICP Winlab Software Guide, Perkin Elmer. Part No. 0993-8966.

12.3 Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma- Atomic Emission Spectrometry, Method 200.7, EPA/600/R-04/111 May 1994.

12.4 Environmental Express procedure for Lead Analysis with the Ghost Wipe, per HUD Guidelines for Lead in Dust Wipes, Appendix A-5.0 and NIOSH Standard 7082.

12.5 NELAC/NELAP, Chapter 5, June 2003

12.6 DoD Quality Systems Manual, Final Revision 4.2, October 2010

12.7 MCP WSC-CAM Section IIIA.

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13.0 POLLUTION PREVENTION and WASTE MANAGEMENT

13.1 ESS Laboratory's policies on pollution prevention and waste management are covered in SOP 90_0002, Hazardous Waste Contingency and Emergency Response Plan. All employees are trained in the requirements of the SOP.

14.0 METHOD PERFORMANCE

14.1 Precision and Accuracy data must be generated by all employees before performing this analysis on client samples. The data is generated by analyzing a method blank and four blank spike samples. Acceptance criteria are 85-115% Recovery and %RSD of ≤ 20%.

14.2 The precision and accuracy data in Table 1 are typical for aqueous metals analysis.

15.0 TABLES, DIAGRAMS, ATTACHMENTS, AND VALIDATION DATA

Table 1. Typical Precision and Accuracy data generated 2/14/2005

Compound Name	Spk	Avg	%Rec	%RSD	Compound Name	Spk	Avg	%Rec	%RSD
Silver	0.25	0.2409	96.4	3	Manganese	0.5	0.4863	97.3	4
Aluminum	2.5	2.5089	100.4	4	Molybdenum	0.5	0.4919	98.4	4
Arsenic	0.5	0.4615	92.3	5	Sodium	25	24.0496	96.2	4
Boron	0.5	0.4843	96.9	4	Nickel	0.5	0.4991	99.8	4
Barium	0.5	0.4803	96.1	4	Lead	0.5	0.4856	97.1	4
Beryllium	0.05	0.0489	97.8	4	Antimony	0.5	0.4652	93.0	3
Calcium	5	4.8144	96.3	4	Selenium	1	0.9435	94.3	4
Cadmium	0.25	0.2258	90.3	4	Tin	0.5	0.4809	96.2	4
Cobalt	0.5	0.4838	96.8	4	Strontinum	0.05	0.0490	98.0	4
Chromium	0.5	0.4878	97.6	4	Titanium	0.5	0.4845	96.9	4
Copper	0.5	0.5010	100.2	4	Thallium	0.5	0.4727	94.5	5
Iron	2.5	2.4523	98.1	4	Vanadium	0.5	0.5030	100.6	4
Potassium	25	22.0020	88.0	4	Zinc	0.5	0.4614	92.3	4
Mangnesium	5	4.7553	95.1	4					

Attachments:

Table 2 DoD QSM BS/MS QC Limits

Table 3 Instrument Wavelengths and Limits

Table 4 General Method Quality Objectives

Attachment 1 Method of Standard Additions

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Attachment A-1	Deleted.
Attachment A-2	Deleted.
Attachment B-1	ICP II Tray Sequence Logbook
Attachment B-2	ICP III Tray Sequence Logbook
Attachment C	Metals Data Table
Attachment D	DOD QSM Version 4.2 Method Quality Objectives.
Attachment E	MCP WSC-CAM Section IIIA Method Quality Objectives.

16.0 DEFINITIONS

16.1 **Calibration Blank (CB)** -- A volume of reagent water fortified with the same matrix as the calibration standards, but without the analytes.

16.2 **Calibration Standard (CAL)** -- A solution prepared from the primary dilution standard solution or stock standard solutions. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

16.3 **Blank spike (BS, LCS)** -- An aliquot of reagent water or other blank matrices to which known quantities of the method analytes are added in the laboratory. The BS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. When it is necessary to test for lead, per HUD specifications, the LCS is prepared by spiking a Ghost Wipe (ASTM E 1792, current lot) with an appropriate amount of ICV solution.

16.4 **Matrix spike (MS)** -- An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The MS is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS corrected for background concentrations.

16.5 **Matrix spike duplicate (MSD)** -- An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The MSD is analyzed exactly like a sample, and its purpose is to determine the precision of the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MSD corrected for background concentrations.

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16.6 **Method Blank (MB)** -- An aliquot of reagent water or other blank matrices that are treated exactly as a sample including exposure to all glassware, equipment, and reagents that are used with other samples. The MB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

16.7 **Method Detection Limit (MDL)** -- The minimum concentration of an analyte that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero.

16.8 **Quality Control Sample (QCS)** -- A solution of method analytes of known concentrations that is used to fortify an aliquot of BS or sample matrix. The QCS is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check laboratory performance with externally prepared test materials.

16.9 **Stock Standard Solution (SSS)** -- A concentrated solution containing one or more method analytes prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source.

17.0 PERSONNEL QUALIFICATIONS

17.1 Analysts who perform this analysis must have a working knowledge or quantitative and qualitative analysis, instrumental methods of analysis, chemical laboratory methods, and equipment.

17.2 All analysts, before performing any analysis, participate in the ESS Laboratory training program (SOP 80_0016). The training process consists of reading the Standard Operating Procedure, gaining instruction on the procedure from an experienced analyst, and performing the initial demonstration of capability.

18.0 TROUBLESHOOTING/MAINTENANCE

18.1 **Instrument Maintenance**:

18.1.1 **Daily:**

- Inspect water level in re-circulating water chiller and inspect for leaks.
- Inspect peristaltic pump tubing, replace if worn.
- Inspect RF coil for excess condensation, wipe down to avoid arcing. Also, the coil should be cool to the touch, which indicates that cooling water is circulating through system.
- Inspect purge window and clean as needed.

18.1.2 **Monthly**

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- Check ventilation filters clean as needed.

18.1.3 **Bi-annually**

- Replace chiller water.
- Vacuum ICP air vents

18.2 Record all maintenance in the instrument's maintenance logbook.

18.3 Extraordinary problems may require assistance from Perkin-Elmer Corporation. ESS Laboratory maintains a service contract with this vendor.

19.0 Data Management And Records

19.1 **Data Management** - ESS Laboratory's utilizes the Promium Element LIMS system as part of its Data Management system. Client sample information is entered into ELEMENT LIMS and analyses are assigned to each sample. The LIMS allows EPA hold times, minimum batch QC requirements, and QC criteria to be assigned to each analysis. Standards can be entered and assigned to QC samples through the LIMS. Once analysis has been performed, data is imported using DataTool avoiding manual errors. In conjunction with Crystal Reports, the ELEMENT system allows for a wide variety of reporting formats.

19.2 **Records** – The specific retention periods required in the NELAC Standards, EPA-CFR and state and local statutes are followed or exceeded. At a minimum, data records are retained for five years from last use (10 years for drinking water). If there is a question about whether a record should be retained or disposed because no specific requirement could be found, the record is retained until such time as a retention period is specified. Records are stored in specified-labeled locations and are easily retrievable. All raw data associated with testing is also retained including; computer printouts, chromatograms, review forms, and logbooks.

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Table 2 DoD Quality Systems Manual Blank Spike / Matrix Spike QC Limits

	DoD		
Aqueous			
Analyte	LCL	UCL	LMEL
Silver	80	120	75
Aluminum	80	120	80
Arsenic	80	120	80
Barium	80	120	80
Beryllium	80	120	80
Calcium	80	120	80
Cadmium	80	120	80
Cobalt	80	120	80
Chromium	80	120	80
Copper	80	120	80
Iron	80	120	80
Potassium	80	120	80
Mangnesium	80	120	80
Manganese	80	120	80
Molybdenum	80	120	75
Sodium	80	120	80
Nickel	80	120	80
Lead	80	120	80
Antimony	80	120	80
Selenium	80	120	75
Thallium	80	120	80
Vanadium	80	120	80
Zinc	80	120	80
Solid			
Silver	75	120	70
Aluminum	80	120	75
Arsenic	80	120	80
Barium	80	120	80
Beryllium	80	120	80
Calcium	80	120	80
Cadmium	80	120	80
Cobalt	80	120	80
Chromium	80	120	80
Copper	80	120	80
Iron	80	120	80
Potassium	80	120	80
Mangnesium	80	120	80
Manganese	80	120	80
Molybdenum	80	120	75
Sodium	80	120	80
Nickel	80	120	80
Lead	60	130	60
Antimony	80	120	75
Selenium	80	120	75
Thallium	80	120	80
Vanadium	80	120	80
Zinc	80	120	80

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TABLE 3

INSTRUMENT WAVELENGTHS AND LIMITS

Detection Element	Wavelength(nm)
Aluminum	237.313
Antimony	206.836
Arsenic	188.979
Barium	233.527
Beryllium	313.107
Boron	182.528
Cadmium	228.802
Calcium	315.886
Chromium	267.716
Cobalt	228.616
Copper	324.752
Iron	238.204
Iron	302.107
Lead	220.353
Magnesium	279.077
Manganese	257.610
Molybdenum	202.031
Nickel	231.604
Potassium	766.491
Selenium	196.026
Silver	328.068
Sodium	330.237
Thallium	190.801
Tin	189.927
Titanium	337.279
Vanadium	292.402
Zinc	213.857

The wavelengths listed are utilized because of their sensitivity and overall acceptance. Other wavelengths may be substituted if they can provide the needed sensitivity and are treated with the same corrective techniques for spectral interference (see step 5.1).

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Table 4

Summary of Method Quality Objectives for Method 200.7/6010B
Metals by Inductively Coupled Plasma

QC Element	Frequency	Criteria	Corrective Action
Initial Calibration	Daily following optimization of ICP and prior sample analysis.	• Minimum of blank and three standards. • Low standard at MRL • R ≥0.995 (Do not force through zero for LR)	• No allowance. Perform maintenance and recalibrate.
ICV	Immediately following daily initial calibration.	• %Rec = 90-110%, %RPD < 5%. • Use separate source from initial calibration standards.	• If criteria exceeded, remake and re-analyze ICV. If second consecutive ICV is within criteria then calibration is accepted, otherwise recalibrate.
Low Level Calibration Standard (CRI)	Daily after initial calibration to support MRL.	• Only used if low standard is not at the MRL. • %Recovery 70-130%. • MRL is at level of last successfully passed CRL.	• If no CRL passes, MRL at concentration in lowest calibration standard.
CCV	After calibration, every 10 samples and at end of analytical run.	• Concentration level near midpoint of curve • Same source as calibration standard. • %Rec = 90-110%, %RPD ≤5% (200.9 first CCV 95-105%)	• If criteria are exceeded, then remake and re-analyze CCV. If second consecutive CCV is within criteria, then calibration is verified, otherwise re-calibrate system and re-analyze any sample since the last valid CCV.
Continuing Calibration Blank	After calibration, every 10 samples and at end of analytical run.	• Must be matrix-matched (same acid concentration as standards and QC samples.) • Analytes < MRL	• Re-calibrate and re-analyze all samples since last valid CCB.
Method Blank	One per analytical batch of 20 or fewer samples.	• Matrix specific • Analytes < MRL	• Report exceedance in the project narrative. • Samples that are non-detect may be reported. • Samples with concentrations that are 20x higher than the method blank may be reported. • Samples reported with a contaminated blank must be "B" flagged.
Blank spike (BS)	One per analytical batch of 20 or fewer samples.	• Use standard source different than used for initial calibration and Matrix specific • Concentration level should be between low and mid-level standard • Percent recoveries 80-120%. (85-115% for 200.7)	• Report exceedance in the project narrative. • If LCS is biased high and sample is non-detect, then may report sample result. • Re-digest and re-analyze if the above exceptions do not apply.

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Blank spike duplicate (BSD)	One per analytical batch of 20 or fewer samples.	• Prepared as above • Percent recoveries same as above • %RPD ≤ 20% aqueous and ≤ 30% soil.	• Report exceedance in the project narrative. • If LCS is biased high and sample is non-detect, then may report sample result. • Re-digest and re-analyze if the above exceptions do not apply.
Matrix Spike	One per analytical batch of 10 or fewer samples	• Prepared using the same source as the blank spike • Concentration level should be between low and mid-level standard • Percent recoveries between 75-125%.	• Report exceedance in the project narrative. • Laboratories are expected to develop in-house control limits per each media. Control limits should fall within default limits.
Matrix duplicate	One per analytical batch of 10 or fewer samples	• **Aqueous**: Relative percent difference is ±20% for samples > 5x MRL and ±MRL for samples <5x the MRL. • **Soil**: Relative percent difference is ±35% for samples > 5x MRL and ±2xMRL for samples <5x the MRL.	• Note exceedance in project narrative. • If MS %Recovery is >30% and LCS is in control, no corrective action is required. If %Recovery is <30% and non-detect results found perform PDS.
Serial Dilution	One per analytical batch of 10 or fewer samples, performed on Dup/Spk sample.	• Result of 5x dilution must be ±10% of undiluted sample when result is ≥25x MRL.	• Narrate serial dilution QC out of control. May be indication of matrix affect.
Post Digestion Spike (PDS)	One per analytical batch of 10 or fewer samples, performed on Dup/Spk sample.	• Perform PDS if Serial dilution is not in control or if sample is < 25x the MRL. • Percent recovery must be 85-115% (75-125% for 6010B).	• Narrate PDS out of control if MS is also out of control. May be indication of matrix affect.
Linear Dynamic Range	Semi-annually	• Use successively higher concentrated standards against calibration curve until a standard is reached that is >10% difference from true value. Upper LDR is 90% of the highest standard within criteria.	• N/A
Instrument Detection Limit	Quarterly	• Analyze ten replicates of the calibration blank.	• N/A

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ATTACHMENT I

SOP Modification

Method of Standard Additions

SOP(s) Modified: Inductively Coupled Plasma-Atomic Emission Spectroscopy (SW 846 Method 6010B/ EPA Method 200.7

Modification Objective: This addendum defines when the method of standard additions must be performed and how to perform the procedure.

Method Summary: The standard addition technique involves adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample constituent that enhances or depresses the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences which cause a baseline shift.

For DoD/Navy projects, the method of standard additions (MSA) shall be used when the matrix spike or matrix spike duplicate is outside control limits and the failure is confirmed by the serial dilution/post digestion spike test. MSA calculation will only be applied to the metal(s) that failed.

MSA Procedure: The simplest version of this technique is the single-addition method, in which two identical aliquots of the sample solution, each of volume V_x, are taken. To the first (labeled A) is added a known volume V_S of a standard analyte solution of concentration C_S. To the second aliquot (labeled B) is added the same volume Vx of the solvent. The analytical signals of A and B are measured and corrected for non-analyte signals (performed by the ICP). The unknown sample concentration C_X is calculated:

$$C_X = \frac{S_B \times V_S \times C_S}{(S_A - S_B) \times V_x}$$

Where S_A and S_B are the analytical signals of solutions A and B, respectively. V_S and C_S should be chosen so that S_A is roughly twice S_B on the average, avoiding excess dilution of the sample.

Since ESS Laboratory performs a post digestion spike (PDS) on each sample it has chosen for the matrix spike the results from the spiked and unspiked aliquots will be used in the MSA calculation.

Example: MS result for Titanum was 56.8% and the PDS was at 69%. MSA calculated as follows:

$V_x = 10$ ml $V_S = 0.05$ ml $C_S = 100$ mg/L

Signal S_A (spiked aliquot) = 109644
Signal S_B (unspiked aliquot) = 19801

$$Cx = \frac{(19801 \times 0.05 \text{ ml} \times 100 \text{ mg/L})}{(109644 - 19801) \times 10 \text{ ml}} = 0.11 \text{ mg/L}$$

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ATTACHMENT B-1

ESS LABORATORY
ICP II TRAY SEQUENCE LOGBOOK

#	SAMPLE	#	SAMPLE	#	SAMPLE	#	SAMPLE
1	STD 1:	31		61		91	
2	STD 2:	32		62		92	
3	STD 3:	33		63		93	
4	STD 4:	34		64		94	
5	SCV1:	35		65		95	
6	CRL 1:	36		66		96	
7	CRL 2:	37		67		97	
8	CRL 3:	38		68		98	
9		39		69		99	
10		40		70		100	
11		41		71		101	
12		42		72		102	
13		43		73		103	
14		44		74		104	
15		45		75		105	IFB:
16		46		76		106	IFA:
17		47		77			
18		48		78			
19		49		79			
20		50		80			
21		51		81			
22		52		82			
23		53		83			
24		54		84			
25		55		85			
26		56		86			
27		57		87			
28		58		88			
29		59		89			
30		60		90			

Internal Standard

ID:_____

SIF:_____

RDS:_____

METHOD:_____

ANALYST:_____

DATE:_____

Comments:_____

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ATTACHMENT B-2

ESS LABORATORY
ICP III TRAY SEQUENCE LOGBOOK

#	SAMPLE	#	SAMPLE	#	SAMPLE	#	SAMPLE
1	STD 1:	31		61		91	
2	STD 2:	32		62		92	
3	STD 3:	33		63		93	
4	STD 4:	34		64		94	
5	SCV1:	35		65		95	
6	CRL 1:	36		66		96	
7	CRL 2:	37		67		97	
8	CRL 3:	38		68		98	
9		39		69		99	
10		40		70		100	
11		41		71		101	
12		42		72		102	
13		43		73		103	
14		44		74		104	
15		45		75		105	IFB:
16		46		76		106	IFA:
17		47		77			
18		48		78			
19		49		79			
20		50		80			
21		51		81			
22		52		82			
23		53		83			
24		54		84			
25		55		85			
26		56		86			
27		57		87			
28		58		88			
29		59		89			
30		60		90			

Internal Standard

ID:_____

SIF:_____

RDS:_____

METHOD:_____

ANALYST:_____

DATE:_____

Comments:_____

CONTROL# 30.0038-1001A PAGE____

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Cranston, RI

ATTACHMENT C

Procedure: 30_6010B R.8
Inductively Coupled Plasma

Metals Data Table

ESS Project ID _____

ESS Sample ID	Element(s)	Dilution	File ID	1st Reviewer

Comments: _____

	Aqueous ICP	Aqueous GFAA	Aqueous Hg	Cr^{+6}	TCLP Hg SPLP Hg	TCLP/SPLP Metals
Entered						
Reviewed						

	Soil ICP	Soil GFAA	Soil Hg	Cr^{+6}	TCLP Hg SPLP Hg	TCLP/SPLP Metals
Entered						
Reviewed						

I certify that I have read, understood, and implemented the requirements of the SOP relating to this document.

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Table F-7. Inorganic Analysis by Inductively Coupled Plasma (ICP) Atomic Emission Spectrometry and Atomic Absorption Spectrophotometry (AA) (Methods 6010 and 7000 Series)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see Section C.1.f).	NA.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification (See Box D-13)					
LOQ establishment and verification (See Box D-14)					
Instrument detection limit (IDL) study (ICP only)	At initial set-up and after significant change in instrument type, personnel, test method, or sample matrix.	IDLs shall be ≤ LOD.	NA.	NA.	Samples may not be analyzed without a valid IDL.
Linear dynamic range or high-level check standard (ICP only)	Every 6 months.	Within ± 10% of true value.	NA.	NA.	

Table F-7. Inorganic Analysis by Inductively Coupled Plasma (ICP) Atomic Emission Spectrometry and Atomic Absorption Spectrophotometry (AA) (Methods 6010 and 7000 Series) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial calibration (ICAL) for all analytes **ICP: minimum one high standard and a calibration blank;** **GFAA: minimum three standards and a calibration blank;** **CVAA: minimum 5 standards and a calibration blank**	Daily ICAL prior to sample analysis.	If more than one calibration standard is used, $r \geq 0.995$.	Correct problem, then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed.
Second source calibration verification (ICV)	Once after each ICAL, prior to beginning a sample run.	Value of second source for all analyte(s) within ± 10% of true value.	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
Continuing calibration verification (CCV)	After every 10 field samples and at the end of the analysis sequence.	ICP: within ± 10% of true value; GFAA: within ± 20% of true value; CVAA: within ± 20% of true value.	Correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Low-level calibration check standard (ICP only)	Daily, after one-point ICAL.	Within ± 20% of true value.	Correct problem, then reanalyze.	Flagging criteria are not appropriate.	No samples may be analyzed without a valid low-level calibration check standard. Low-level calibration check standard should be less than or equal to the reporting limit.

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Table F-7. Inorganic Analysis by Inductively Coupled Plasma (ICP) Atomic Emission Spectrometry and Atomic Absorption Spectrophotometry (AA) (Methods 6010 and 7000 Series) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method blank	One per preparatory batch.	No analytes detected > ½ RL and greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common laboratory contaminants, no analytes detected > RL (see Box D-1).	Correct problem, then see criteria in Box D-1. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Calibration blank	Before beginning a sample run, after every 10 samples, and at end of the analysis sequence.	No analytes detected > LOD.	Correct problem. Re-prep and reanalyze calibration blank. All samples following the last acceptable calibration blank must be reanalyzed.	Apply B-flag to all results for specific analyte(s) in all samples associated with the blank.	
Interference check solutions (ICS) (ICP only)	At the beginning of an analytical run.	ICS-A: Absolute value of concentration for all non-spiked analytes < LOD (unless they are a verified trace impurity from one of the spiked analytes); ICS-AB: Within ± 20% of true value.	Terminate analysis; locate and correct problem; reanalyze ICS, reanalyze all samples.	If corrective action fails, apply Q-flag to all results for specific analyte(s) in all samples associated with the ICS.	
LCS containing all analytes to be reported	One per preparatory batch.	QC acceptance criteria specified by DoD, if available; see Box D-3 and Appendix G.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available (see full explanation in Appendix G).	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

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Table F-7. Inorganic Analysis by Inductively Coupled Plasma (ICP) Atomic Emission Spectrometry and Atomic Absorption Spectrophotometry (AA) (Methods 6010 and 7000 Series) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix spike (MS)	One per preparatory batch per matrix (see Box D-7).	For matrix evaluation, use QC acceptance criteria specified by DoD for LCS.	Examine the project-specific DQOs. If the matrix spike falls outside of DoD criteria, additional quality control tests are required to evaluate matrix effects.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix (see Box D-7).	MSD: For matrix evaluation use QC acceptance criteria specified by DoD for LCS. MSD or sample duplicate: RPD ≤ 20% (between MS and MSD or sample and sample duplicate).	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Dilution test (ICP and GFAA only)	One per preparatory batch.	Five-fold dilution must agree within ± 10% of the original measurement.	ICP: Perform post-digestion spike (PDS) addition; GFAA: Perform recovery test.	Flagging criteria are not appropriate.	Only applicable for samples with concentrations > 50 x LOQ.
Post-digestion spike (PDS) addition (ICP only)	When dilution test fails or analyte concentration in all samples < 50 x LOD.	Recovery within 75-125% (see Table B-1).	Run all associated samples in the preparatory batch by method of standard additions (MSA) or see flagging criteria.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	Spike addition should produce a concentration of 10 – 100 x LOQ.
Recovery test (GFAA only)	When dilution test fails or analyte concentration in all samples < 25 x LOD.	Recovery within 85-115%.	Run all associated samples in the preparatory batch by method of standard additions (MSA) or see flagging criteria.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	
Method of standard additions (MSA)	When matrix interference is confirmed.	NA.	NA.	NA.	Document use of MSA in the case narrative.
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

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Table III A-1: Specific QC Requirements and Performance Standards for Metals (SW-846 6010C) Using WSC-CAM-III A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
Initial Demonstration of Proficiency (IDP)	Laboratory Analytical Accuracy & Precision	(1) Must be performed prior to using method on samples. (2) Must be performed for each matrix. (3) Must contain all target analytes. (4) Must follow procedures in Section 9.4 of SW-846 6010C and the applicable preparation method (SW-846 3000 series).	No	NA	Refer to Section 9.4 of SW-846 6010C, the applicable preparation method requirements in SW-846 3000 series methods, and Section 1.1.2 of this protocol.	NA
Preparation of Samples	Accuracy and Representativeness	(1) All aqueous (except dissolved/filtered groundwaters) and solid samples must be prepared (digested) prior to analysis. See Appendix III A-4 for preparation method references.	No	NA	NA	NA
Linear Dynamic Range (LDR)	Laboratory Analytical Accuracy	(1) Frequency: check LDR every 6 months (Section 10.4 of SW-846 6010C). (2) Determine the upper limit of the linear dynamic range for each wavelength by determining the signal responses from a minimum of 3 different concentration standards across the range. See SW-846 Method 6010C for details.	No	NA	NA	NA
Initial Calibration	Laboratory Analytical Accuracy	(1) Frequency: Following profiling and optimization of ICP; daily prior to sample analysis. (2) Minimum calibration blank plus one calibration standard for each target analyte or a multi-point curve. (3) Linear regression with correlation coefficient $r \geq 0.998$; non-linear regression may be used if $r^2 \geq 0.998$.	No	NA	Perform instrument maintenance as necessary; re-optimize instrument; re-calibrate as required by SW-846 6010C.	Suspend all analyses until initial calibration meets criteria.
Initial Calibration Verification (ICV)	Laboratory Analytical Accuracy	(1) Frequency: Immediately after each initial calibration. (2) Prepared using standard source different than used for initial calibration. (3) Concentration level near midpoint of curve. (4) Must contain all target analytes.	No	NA	(1) Reanalyze ICV; if acceptable, no further action required. (2) If reanalysis is still outside of criteria, recalibrate and reanalyze ICV.	Suspend all analyses until ICV meets criteria.

Table III A-1: Specific QC Requirements and Performance Standards for Metals (SW-846 6010C) Using WSC-CAM-III A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
		(5) Percent recoveries must be between 90-110% for each target analyte.				
Initial Calibration Blank (ICB)	Laboratory Analytical Sensitivity (instrument drift & contamination)	(1) Frequency: Immediately after ICV. (2) Prepared using same concentration of acids as calibration standards. (3) Target analytes must be <RL.	No	NA	(1) Reanalyze ICB; if acceptable, no further action required. (2) If reanalysis is still outside of criteria, recalibrate and reanalyze ICV & ICB.	Suspend all analyses until ICB meets criteria.
Low-Level Calibration Verification (LLCV)	Laboratory Analytical Sensitivity (verify low-end of calibration range / verify RL)	(1) Frequency: Daily prior to sample analysis if initial calibration did not contain a low-level standard at the RL for each target analyte. If initial calibration includes the RL as the low-level standard in the initial calibration curve, then LLCV is not required. (2) Prepared using same source as initial calibration standards. (3) Concentration level must be at the level of the RL for all target analytes. (4) Percent recoveries must be 70-130% for all target analytes.	No	NA	(1) Reanalyze LLCV; if acceptable, no further action required. (2) If reanalysis is still outside of criteria and associated analytes are ≤10x RL in associated field samples, recalibrate and reanalyze LLCV and associated samples. (3) If associated analytes are >10x RL in associated field samples, include explanation in laboratory narrative; no further action required.	Suspend all analyses until LLCV meets criteria unless the concentrations of the affected target analytes are >10x RL in the associated field samples.
Interference Check Standards (ICSA and ICSAB)	Laboratory Analytical Accuracy (checks background points and interelement interference corrections on instrument)	(1) Frequency: Daily prior to sample analysis. (2) ICSA and ICSAB must contain known amounts of interfering analytes (see SW-846 6010C). (3) Percent recoveries must be 80-120% for all target analytes. (4) Non-spiked analytes in the ICSA must be <2x RL.	No	NA	(1) Reanalyze ICSA/AB; if acceptable, no further action required. (2) If ICSA/AB is still outside of criteria, adjust interference corrections, background corrections, and/or linear ranges, as needed and reanalyze ICSA/AB. (3) Recalibrate and reanalyze all samples since last compliant ICSA/AB.	Suspend all analyses until ICSA/AB meet criteria. If automatic (computerized) corrections for background and IECs are not used during analysis, the laboratory must narrate how spectral interferences were minimized and what hand-calculations, if any, were performed to correct sample results.

Table III A-1: Specific QC Requirements and Performance Standards for Metals (SW-846 6010C) Using WSC-CAM-III A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
Continuing Calibration Verification (CCV)	Laboratory Analytical Accuracy	(1) Frequency: Every 10 samples and at the end of the analytical run. (2) Prepared using same source as initial calibration standards. (3) Concentration level near midpoint of curve. (4) Must contain all target analytes. (5) Percent recoveries must be 90-110% for each target analyte.	No	NA	(1) Reanalyze CCV; if acceptable, no further action required. (2) If reanalysis is still outside of criteria, recalibrate and reanalyze all associated samples since last compliant CCV – unless (3) applies. (3) If recovery is high (>110%) and all associated sample results are non-detected, no corrective action required.	If (3) applies, include explanation in laboratory narrative.
Continuing Calibration Blank (CCB)	Laboratory Analytical Sensitivity (instrument drift & contamination)	(1) Frequency: Every 10 samples following CCV and at the end of the analytical run. (2) Prepared using same concentration of acids as calibration standards. (3) Target analytes must be <RL.	No	NA	(1) Reanalyze CCB; if acceptable, no further action required. (2) If reanalysis is still outside of criteria, recalibrate and reanalyze all associated samples since last compliant CCB – unless (3) applies. (3) If concentration of contaminant in CCB is >RL but all associated sample results are either non-detected or >10x concentration in CCB, no corrective action required.	If (3) applies, include explanation in laboratory narrative.

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Table III A-1: Specific QC Requirements and Performance Standards for Metals (SW-846 6010C) Using WSC-CAM-III A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
Method Blank (MB)	Laboratory Method Sensitivity (contamination evaluation)	(1) Frequency: One per digestion batch of ≤20 field samples. (2) Must be digested with the samples using the same preparation method as the samples. (3) Target analytes must be <RL.	Yes	NA	(1) Reanalyze MB; if acceptable, no further action required. (2) If reanalysis is still outside of criteria, redigest and reanalyze MB and all associated field samples in batch – unless (3) applies. (3) If concentration of contaminant in MB is >RL but all associated sample results are either non-detected or >10x concentration in MB, no corrective action required.	If (3) applies, include explanation in laboratory narrative.
Laboratory Control Sample (LCS)	Laboratory Analytical Accuracy	(1) Frequency: One per digestion batch of ≤20 field samples. (2) Must be matrix-matched by digesting with the samples using the same preparation method. CAM requires a solid Standard Reference Material (SRM) be prepared and analyzed with solid field samples as the "solid LCS." An SRM is a soil or sediment matrix that contains the analytes of interest at known concentrations and with 95% confidence limits. (3) Concentration levels for aqueous LCS near midpoint of curve. (4) Must contain all target analytes. (5) Percent recoveries for all target analytes must be 80-120% for aqueous LCS and within vendor control limits (95% confidence limits) for solid LCS.	Yes	Aqueous LCS: Recovery <50%: affected analytes in associated samples may be rejected.	(1) Reanalyze LCS; if acceptable, no further action required. (2) If reanalysis is still outside of criteria and LCSD is in-control for same analyte, no corrective action required. (3) If LCS and LCSD are both outside of criteria, redigest and reanalyze LCS/LCSD and all associated field samples in batch.	Report recovery exceedances in laboratory narrative.

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Table III A-1: Specific QC Requirements and Performance Standards for Metals (SW-846 6010C) Using WSC-CAM-III A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
LCS Duplicate (LCSD)	Laboratory Analytical Accuracy & Precision	(1) Frequency: One per digestion batch of ≤20 field samples ONLY if not performing project-specific MD. (2) Must be matrix-matched by digesting with the samples using the same preparation method. CAM requires a solid SRM be prepared and analyzed with solid field samples as the "solid LCSD." An SRM is a soil or sediment matrix that contains the analytes of interest at known concentrations and with 95% confidence limits. (3) Concentration levels must be same as LCS. (4) Must contain all target analytes; analyze immediately following LCS. (5) Percent recoveries for all target analytes must be 80-120% for aqueous LCS and within vendor control limits (95% confidence limits) for solid LCS. (6) RPDs must be ≤20 for aqueous LCS/LCSD and ≤30 for solid LCS/LCSD.	Yes ONLY if no MD	Same as above for LCS for recovery evaluation	(1) Reanalyze LCSD; if acceptable, no further action required. (2) If reanalysis is still outside of recovery criteria and LCS is in-control for same analyte, no corrective action required. (3) If LCSD and LCS are both outside of recovery criteria, redigest and reanalyze LCS/LCSD and all associated field samples in batch.	Report recovery and RPD exceedances in laboratory narrative.
Matrix Spike (MS) Project-Specific	Method Accuracy in Sample Matrix	(1) Solid Samples (Soil/Sediment) Frequency: One per 20 field samples per matrix; designated by data user on COC or at project set-up. Aqueous Samples Frequency: One per digestion batch of ≤20 field samples per matrix strongly recommended (designated by data user on COC or at project set-up). (2) Concentration levels near midpoint of curve. (3) Must contain all target analytes. (4) Percent recoveries for all target analytes must be 75-125%.	Yes ONLY when requested by the data user	Recovery <30%: affects non-detects for affected metal in all associated samples.	(1) Reanalyze MS; if acceptable, no further action required. (2) After reanalysis, if MS recovery is 30-74% or >125% and LCS was in-control, no corrective action is required. (3) If MS recovery is <30% and associated with non-detected results, redigest (homogenize sample well) and reanalyze sample/MS pair. Report results and narrate.	Report MS exceedances in laboratory narrative. If redigested due to recoveries <30%, report both sets of sample/MS data.

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Table III A-1: Specific QC Requirements and Performance Standards for Metals (SW-846 6010C) Using WSC-CAM-III A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
Matrix Duplicate (MD) Project-Specific	Method Precision in Sample Matrix	(1) Frequency: One per digestion batch of ≤20 field samples per matrix is strongly recommended (designated by data user on COC or at project set-up). (2) Prepare by digesting and analyzing an additional aliquot of the same field sample used for MS. (3) RPD for each target analyte must be ≤20 for aqueous and ≤35 for solids.	Yes ONLY when requested by the data user	NA	Narrate.	Report exceedances in laboratory narrative.
Dilution Test	Accuracy in Sample Matrix	(1) Frequency: One per ≤20 field samples per matrix; only if project-specific MS requested and analyte concentration is >50x RL. (2) Perform 5x serial dilution on same sample used for MS/MD. (3) %D of the Sample & Dilution results for target analytes at levels >50x RL must be ±10% for all matrices.	Yes ONLY if project-specific MS requested by data user	NA	Narrate.	Report exceedances in laboratory narrative.
General Reporting Issues	NA	(1) Non-detected values must be reported with the sample-specific RL for each target analyte using all preparation/dilution factors. (2) The laboratory must only report values ≥ the sample-specific RL; optionally, values below the sample-specific RL can be reported as estimated, if requested. (see SW-846 Method 6010C, Section 10.3.3). The laboratory must report results for samples and blanks in a consistent manner. (3) Sample concentrations that exceed the LDR must be diluted and reanalyzed to fall within the linear dynamic range. (4) Results for soils/sediments must be reported on a dry-weight basis for comparison to MCP regulatory standards.	NA	NA	NA	(1) Qualification of the data is required if reporting values below the sample-specific RL. (2) The performance of dilutions must be documented in the laboratory narrative or on the report form. Unless due to elevated concentrations of target analytes, reasons for dilutions must be explained in the laboratory narrative. (3) If samples are not preserved properly or are not received with an

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Nitrate

DOC316.53.01069

Cadmium Reduction Method **Method 8171**

MR (0.1 to 10.0 mg/L NO_3^-–N) **Powder Pillows or AccuVac® Ampuls**

Scope and Application: For water, wastewater and seawater

Test preparation

How to use instrument-specific information

The *Instrument-specific information* table displays requirements that may vary between instruments. To use this table, select an instrument then read across to find the corresponding information required to perform this test.

Table 244 Instrument-specific information

Instrument	Powder pillows		AccuVac Ampuls	
	Sample cell	Cell orientation	Sample cell	Adapter
DR 6000	2495402	Fill line faces right	2427606	—
DR 5000	2495402	Fill line faces user	2427606	—
DR 3900	2495402	Fill line faces user	2427606	LZV846 (A)
DR 3800, DR 2800, DR 2700	2495402	Fill line faces right	2122800	LZV584 (C)

Before starting the test:

For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water instead of the sample.

A deposit of unoxidized metal will remain at the bottom of the cell after the NitraVer® 5 dissolves. The deposit will not affect results.

This method is technique-sensitive. Shaking time and technique influence color development. For most accurate results, make successive tests on a 10.0-mg/L Nitrate Nitrogen Standard solution. Adjust shaking times to obtain the correct result.

Rinse the sample cell immediately after use to remove all cadmium particles. Retain the used sample for proper hazardous waste disposal for cadmium.

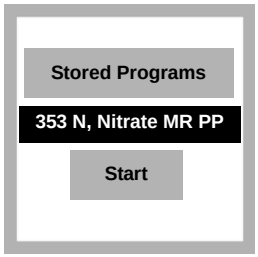
Prepared samples will contain cadmium and must be disposed of according to Federal, State and local hazardous waste regulations. Refer to the current MSDS for safe handling and disposal instructions.

Collect the following items:

Description	Quantity
Powder Pillow Test:	
NitraVer® 5 Nitrate Reagent Powder Pillow	1
Sample Cells (see *Instrument-specific information*)	2
Stopper, Neoprene, #1, solid	2
AccuVac Test:	
NitraVer® 5 Nitrate Reagent AccuVac® Ampul	1
Beaker, 50-mL	1
Sample Cell for blank (see *Instrument-specific information*)	1

See *Consumables and replacement items* for reorder information.

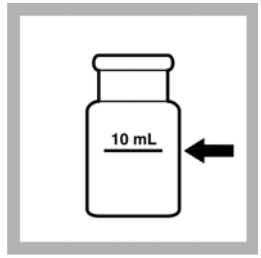
Cadmium reduction method for powder pillows



1. Select the test.

Insert an adapter if required (see *Instrument-specific information*).

Refer to the user manual for orientation.



2. Fill a sample cell with 10 mL of sample.

3. Prepared Sample: Add the contents of one NitraVer 5 Nitrate Reagent Powder Pillow. Insert a stopper into the cell.

4. Start the instrument timer.

A one-minute reaction period will begin.

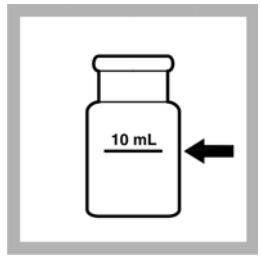
Shake the cell vigorously until the timer expires.
Note: *Some solid material will not dissolve.*

Cadmium reduction method for powder pillows (continued)

5. When the timer expires, start the timer again.

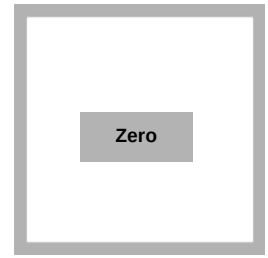
A five-minute reaction period will begin.

An amber color will develop if nitrate is present.



6. Blank Preparation: When the timer expires, fill a second sample cell with 10 mL of sample.

7. Wipe and insert the blank into the cell holder.

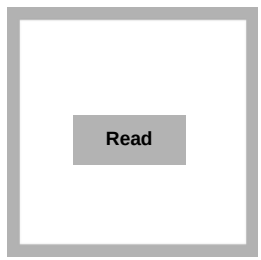


8. **ZERO** the instrument.

The display will show:

0.0 mg/L NO_3^-–N

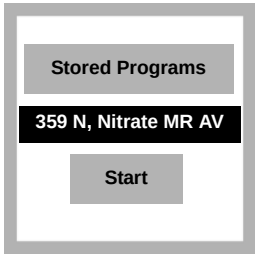
9. Within two minutes after the timer expires, wipe and insert the prepared sample into the cell holder.



10. **READ** the results in mg/L NO_3^-–N.

Refer to the user manual to display other chemical forms.

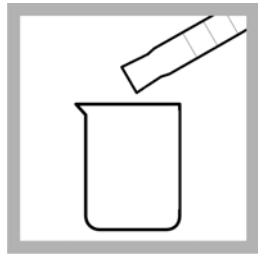
Cadmium reduction method for AccuVac® Ampuls



1. Select the test.

Insert an adapter if required (see *Instrument-specific information*).

Refer to the user manual for orientation.



2. Prepared Sample: Collect at least 40 mL of sample in a 50-mL beaker.

3. Fill a NitraVer 5 Nitrate AccuVac® Ampul with sample. Keep the tip immersed while the Ampul fills completely. Place a stopper over the Ampul tip.

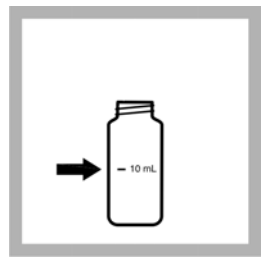
4. Start the instrument timer.

A one-minute reaction period will begin.

5. Invert the Ampul 48–52 times as the timer counts down.

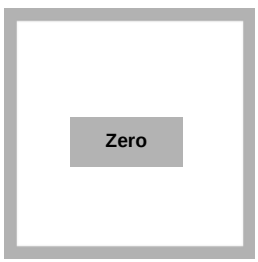
6. When the timer expires, start the timer again.

A five-minute reaction period will begin. An amber color will develop if nitrate is present.



7. Blank Preparation: When the timer expires, fill a round sample cell with 10 mL of sample.

8. Wipe the blank and insert it into the cell holder.

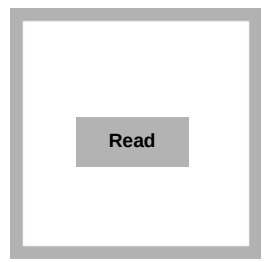


9. **ZERO** the instrument.

The display will show:

0.0 mg/L NO_3^-–N

10. Within two minutes after the timer expires, wipe the Ampul and insert it into the cell holder.



11. **READ** the results in mg/L NO_3^-–N.

Interferences

Table 245 Interfering substances

Interfering substance	Interference level
Chloride	Chloride concentrations above 100 mg/L will cause low results. The test may be used at high chloride concentrations (seawater) but a calibration must be done using standards spiked to the same chloride concentration (see *Seawater calibration*).
Ferric iron	Interferes at all levels
Nitrite	Interferes at all levels Compensate for nitrite interference as follows: **1.** Add 30-g/L Bromine Water[1] drop-wise to the sample until a yellow color remains. **2.** Add one drop of 30-g/L Phenol Solution[1] to destroy the color. **3.** Proceed with Step 2 of the test. Report the results as total nitrate and nitrite.
pH	Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment.
Strong oxidizing and reducing substances	Interfere at all levels

[1] See *Optional reagents and apparatus*.

Seawater calibration

Chloride concentrations above 100 mg/L will cause low results. To perform this test in water with high interference level, calibrate the water using standards spiked to the same chloride concentrations as the required samples. To prepare calibration standards containing 1.0, 3.0, 5.0 and 10.0 mg/L nitrate as NO_3–N:

1. Prepare a 1 L volume of chloride water that matches the concentration of the samples, using the following equation:

   c. Add necessary Chloride concentration (g/L) x (1.6485) = g of ACS grade NaCl to 1 L of deionized water.

   Note: *18.8 g/L is a typical seawater chloride concentration.*

   d. Mix this solution thoroughly to make sure that it is a homogeneous solution. Use this water as the dilution water instead of the deionized water when preparing the nitrate standards.

2. Use Class A glassware or a Tensette Pipet to pipet 1.0, 3.0, 5.0, and 10.0 mL of the 100 mg/L Nitrogen-Nitrate as NO_3^-–N (NIST) Standard Solution (Catalog Number 194749) into four different 100 mL Class A volumetric flasks.
3. Dilute to the mark with the prepared chloride water. Mix thoroughly.
4. Use the prepared chloride water for the 0-mg/L nitrate as NO_3–N standard.

Sample collection, preservation and storage

- Most reliable results are obtained when samples are analyzed as soon as possible after collection. If prompt analysis is impossible, store samples in clean plastic or glass bottles for up to 24 hours at 4 °C. To preserve samples for longer periods, add 2 mL of Concentrated Sulfuric Acid (H_2SO_4)* per liter and store at 4 °C. The results are reported as total nitrate and nitrite.
- Before analysis, warm the sample to room temperature and adjust the pH to 7 with 5.0 N Sodium Hydroxide Standard Solution*. Do not use mercury compounds as preservatives.

* See *Optional reagents and apparatus*.

- Correct the test result for volume additions by dividing the total volume (acid + base + sample) by the original sample volume and multiplying the test result by this factor.

Accuracy check

Standard additions method (sample spike)
Required for accuracy check:

- Nitrate Nitrogen Standard,100-mg/L NO_3^-–N
- TenSette Pipet and Pipet Tips

1. After reading test results, leave the sample cell (unspiked sample) in the instrument.
2. Select **Options>More>Standard Additions** from the instrument menu.
3. Accept the default values for standard concentration, sample volume and spike volumes. After the values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.
4. Open the standard solution bottle.
5. Use the TenSette Pipet to prepare spiked samples: add 0.1 mL, 0.2 mL and 0.3 mL of standard to three 10-mL portions of fresh sample.
6. Follow the *Cadmium reduction method for powder pillows* test procedure for each of the spiked samples, starting with the 0.1 mL sample spike. Measure each of the spiked samples in the instrument.
7. Select **GRAPH** to view the results. Select **IDEAL LINE** (or best-fit) to compare the standard addition results to the theoretical 100% recovery.

Standard additions method for AccuVac Ampuls (sample spike)
Required for accuracy check:

- 500 mg/L Nitrate Nitrogen Ampule Standard Solution
- Ampule breaker
- TenSette Pipet and Pipet Tips
- Mixing cylinder, 50-mL (3)

1. Fill three mixing cylinders each with 50-mL of sample and spike with 0.1 mL, 0.2 mL and 0.3 mL of 500 mg/L Nitrate Nitrogen Ampule Standard Solution.
2. Transfer 40 mL from each of the three mixing cylinders to three 50-mL beakers.
3. Analyze each standard addition sample as described in the *Cadmium reduction method for AccuVac® Ampuls*.
4. Accept each standard additions reading. Each addition should reflect approximately 100% recovery. Standard solution method

Note: *Refer to the instrument user manual for specific software navigation instructions.*

Standard Solution Method
Required for accuracy check:

- 5.0-mg/L Nitrate Nitrogen Standard Solution (prepared)
- 100-mg/L Nitrate Nitrogen Standard
- Deionized water
- 100-mL volumetric flask

- 5-mL Volumetric pipet and pipet filler

  OR

- TenSette Pipet and Pipet Tips

1. Prepare a 5.0-mg/L nitrate nitrogen standard solution as follows:
   a. Pipet 5.0 mL of 100-mg/L Nitrate Nitrogen Standard, into a 100-mL volumetric flask.
   b. Dilute to the mark with deionized water. Mix well.
2. Use this 5.0 mg/L nitrate nitrogen standard solution in place of the sample. Follow the *Cadmium reduction method for powder pillows* test procedure.
3. To adjust the calibration curve using the reading obtained with the standard solution, select **Options>More>Standard Adjust** from the instrument menu.
4. Turn on the Standard Adjust feature and accept the displayed concentration. If an alternate concentration is used, enter the concentration and adjust the curve to that value.

Method performance

Program	Standard	Precision 95% Confidence Limits of Distribution	Sensitivity Concentration change per 0.010 Abs change
353	5.0 mg/L NO_3^-–N	4.8–5.2 mg/L NO_3^-–N	0.04 mg/L NO_3^-–N
359	5.0 mg/L NO_3^-–N	4.6–5.4 mg/L NO_3^-–N	0.05 mg/L NO_3^-–N

Summary of method

Cadmium metal reduces nitrates in the sample to nitrite. The nitrite ion reacts in an acidic medium with sulfanilic acid to form an intermediate diazonium salt. The salt couples with gentisic acid to form an amber colored solution. Test results are measured at 400 nm.

Consumables and replacement items

Required reagents

Description	Quantity/Test	Unit	Catalog number
NitraVer® 5 Nitrate Reagent Powder Pillows (for 10 mL sample)	1	100/pkg	2106169
OR			
NitraVer® 5 Nitrate Reagent AccuVac® Ampul	1	25/pkg	2511025

Required apparatus (powder pillows)

Description	Quantity	Unit	Catalog number
Sample cell, 10 mL square, matched pair	2	2/pkg	2495402
Stopper, Neoprene, solid, size #1	2	12/pkg	1480801

Required apparatus (AccuVac)

Description	Quantity	Unit	Catalog number
Beaker, 50-mL	1	each	50041H
Sample cell, 10 mL round, 25 x 54 mm	1	each	2122800
Sample cell, 10 mL round, 25 x 60 mm	1	6/pkg	2427606

Recommended standards

Description	Unit	Catalog number
Mixed Parameter Drinking Water Standard, for F, NO_3–N, PO_4, SO_4	500 mL	2833049
Nitrate Nitrogen Standard Solution, 100-mg/L NO_3^-–N	500 mL	194749
Nitrate Nitrogen Standard Solution, 500 mg/L NO_3–N, 10-mL ampules	16/pkg	1426010
Water, deionized	4 L	27256

Optional reagents and apparatus

Description	Unit	Catalog number
Ampule breaker for 10 mL ampules	each	2196800
Bromine Water, 30-mg/L	29 mL	221120
Cylinder, mixing, 50 mL	each	2088641
Flask, volumetric, 100-mL	each	1457442
Pipet, TenSette, 0.1–1.0 mL	each	1970001
Pipet Tips, for TenSette Pipet 1970001	50/pkg	2185696
Pipet Tips, for TenSette Pipet 1970001	1000/pkg	2185628
Pipet, volumetric, Class A, 5.0 mL	each	1451537
Pipet, volumetric, Class A, 1.0 mL	each	1451535
Pipet, volumetric, Class A, 3.0 mL	each	1451503
Pipet, volumetric, Class A, 10.0 mL	each	1451538
Pipet Filler, safety bulb	each	1465100
Phenol Solution, 30-g/L	29 mL	211220
5.0 N Sodium Hydroxide Standard Solution	1 L	245053
Sulfuric Acid, concentrated	500 mL	97949

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Edition 7

Nitrite

DOC316.53.01074

USEPA[1] Diazotization **Method 8507**

LR (0.002 to 0.300 mg/L NO_2^-–N) **Powder Pillows or AccuVac® Ampuls**

Scope and Application: For water, wastewater and seawater

[1] USEPA approved for wastewater analysis, Federal Register, 44(85), 25505 (May 1, 1979)

Test preparation

How to use instrument-specific information

The *Instrument-specific information* table displays requirements that may vary between instruments. To use this table, select an instrument then read across to find the corresponding information required to perform this test.

Table 253 Instrument-specific information

Instrument	Powder pillows		AccuVac Ampuls	
	Sample cell	Cell orientation	Sample cell	Adapter
DR 6000	2495402	Fill line faces right	2427606	—
DR 5000	2495402	Fill line faces user	2427606	—
DR 3900	2495402	Fill line faces user	2427606	LZV846 (A)
DR 3800, DR 2800, DR 2700	2495402	Fill line faces right	2122800	LZV584 (C)

Before starting the test:

For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water instead of the sample.

Collect the following items:

Description	Quantity
Powder Pillow Test:	
NitriVer® 3 Nitrite Reagent Powder Pillows	1
Sample Cells (see *Instrument-specific information*)	2
AccuVac Test:	
NitriVer® 3 Nitrite Reagent AccuVac® Ampul.	1
Beaker, 50-mL	1
Sample Cell (see *Instrument-specific information*)	1

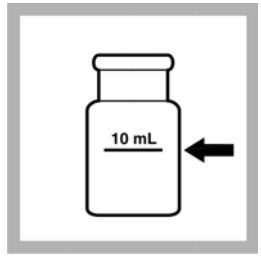
See *Consumables and replacement items* for reorder information.

Diazotization method for powder pillows

1. Select the test.

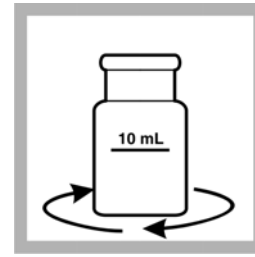
Insert an adapter if required (see *Instrument-specific information*).

Refer to the user manual for orientation.



2. Fill a sample cell with 10 mL of sample.

3. Prepared Sample: Add the contents of one NitriVer 3 Nitrite Reagent Powder Pillow.

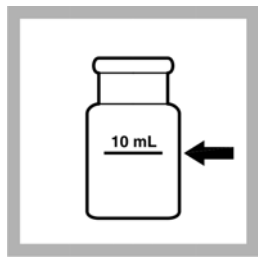


4. Swirl to dissolve.

A pink color will develop if nitrite is present.

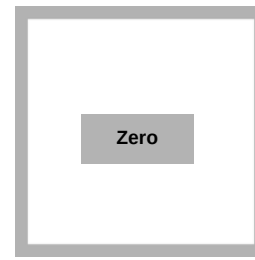
5. Start the instrument timer.

A 20-minute reaction period will begin.



6. Blank Preparation: When the timer expires, fill a second sample cell with 10 mL of sample.

7. Wipe the blank and insert it into the cell holder.

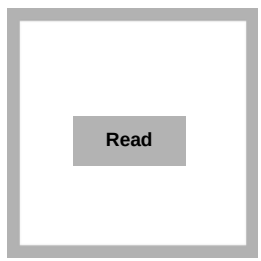


8. ZERO the instrument.

The display will show:

0.000 mg/L NO_2^-–N

9. Wipe the prepared sample and insert it into the cell holder.



10. READ the results in mg/L NO_2^-–N.

Diazotization method for AccuVac® Ampuls

1. Select the test.

Insert an adapter if required (see *Instrument-specific information*).

Refer to the user manual for orientation.

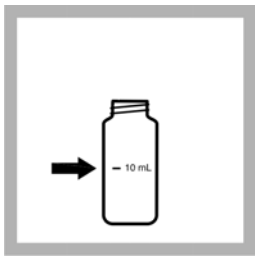
2. Prepared Sample: Collect at least 40 mL of sample into a 50-mL beaker.

Fill a NitriVer 3 Nitrite AccuVac® Ampul with sample. Keep the tip immersed while the Ampul fills.

3. Invert the Ampul several times to mix. A pink color will develop if nitrite is present.

4. Start the instrument timer.

A 20-minute reaction period will begin.



5. Blank Preparation: When the timer expires, fill a sample cell with 10 mL of sample.

6. Wipe the blank and insert it into the cell holder.

ZERO the instrument.

The display will show: 0.000 mg/L NO_2^-–N.

7. Wipe the Ampul and insert it into the cell holder.

READ the results in mg/L NO_2^-–N.

Interferences

Table 254 Interfering substances

Interfering substance	Interference level
Antimonous ions	Interfere by causing precipitation
Auric ions	Interfere by causing precipitation
Bismuth ions	Interfere by causing precipitation
Chloroplatinate ions	Interfere by causing precipitation
Cupric ions	Cause low results
Ferric ions	Interfere by causing precipitation
Ferrous ions	Cause low results
Lead ions	Interfere by causing precipitation
Mercurous ions	Interfere by causing precipitation
Metavanadate ions	Interfere by causing precipitation
Nitrate	Very high levels of nitrate (>100 mg/L nitrate as N) appear to undergo a slight amount of reduction to nitrite, either spontaneously or during the course of the test. A small amount of nitrite will be found at these levels.
Silver ions	Interfere by causing precipitation
Strong oxidizing and reducing substances	Interferes at all levels

Sample collection, preservation and storage

- Collect samples in clean plastic or glass bottles.
- Store at 4 °C (39 °F) or lower if the sample is to be analyzed within 24 to 48 hours.
- Warm to room temperature before running the test.
- Do not use acid preservatives.

Accuracy check

Standard solution method

Note: *Refer to the instrument user manual for specific software navigation instructions.*

1. Preparing nitrite standards is difficult. Use the standard preparation instructions in *Standard Methods for the Examination of Water and Wastewater*, Method 4500—NO_2-B. Prepare a 0.150-mg/L standard.
2. Use the 0.150 mg/L solution in place of the sample. Follow the *Diazotization method for powder pillows* test procedure.
3. To adjust the calibration curve using the reading obtained with the standard solution, select **Options>More>Standard Adjust** from the instrument menu.
4. Turn on the Standard Adjust feature and accept the displayed concentration. If an alternate concentration is used, enter the concentration and adjust the curve to that value.

Method performance

Program	Standard	Precision 95% Confidence Limits of Distribution	Sensitivity Concentration change per 0.010 Abs change
371	0.150 mg/L NO_2^-–N	0.147–0.153 mg/L NO_2^-–N	0.002 mg/L NO_2^-–N
375	0.150 mg/L NO_2^-–N	0.147–0.153 mg/L NO_2^-–N	0.002 mg/L NO_2^-–N

Summary of method

Nitrite in the sample reacts with sulfanilic acid to form an intermediate diazonium salt. This couples with chromotropic acid to produce a pink colored complex directly proportional to the amount of nitrite present. Test results are measured at 507 nm.

Consumables and replacement items

Required reagents

Description	Quantity/Test	Unit	Catalog number
NitriVer® 3 Nitrite Reagent Powder Pillows	1	100/pkg	2107169
OR			
NitriVer® 3 Nitrite Reagent AccuVac® Ampul	1	25/pkg	2512025

Required apparatus

Description	Quantity	Unit	Catalog number
Beaker, 50-mL	1	each	50041H
Sample cell, 10 mL round, 25 x 54 mm	1	each	2122800
Sample cell, 10 mL round, 25 x 60 mm	1	6/pkg	2427606
Sample cell, 10 mL square, matched pair	2	2/pkg	2495402

Recommended standards, reagents and apparatus

Description	Unit	Catalog number
Balance, Analytical, 80 g capacity	each	2936701
AccuVac ampules, for blanks	25/pkg	2677925
AccuVac Snapper	each	2405200
AccuVac Drainer	each	4103600
Handbook, Standard Methods for the Examination of Water and Wastewater	each	2270800
Sodium Nitrite, ACS	454 g	245201
Water, deionized	4 L	27256

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Edition 7

Sulfate

DOC316.53.01135

USEPA[1] SulfaVer 4 Method[2] **Method 8051**

(2 to 70 mg/L) **Powder Pillows or AccuVac® Ampuls**

Scope and Application: For water, wastewater and seawater.

[1] USEPA accepted for reporting wastewater analyses. Procedure is equivalent to USEPA method 375.4 for wastewater.

[2] Adapted from *Standard Methods for the Examination of Water and Wastewater.*

Test preparation

How to use instrument-specific information

The *Instrument-specific information* table displays requirements that may vary between instruments. To use this table, select an instrument then read across to find the corresponding information required to perform this test.

Table 388 Instrument-specific information

Instrument	Powder pillows		AccuVac Ampuls	
	Sample cell	Cell orientation	Sample cell	Adapter
DR 6000	2495402	Fill line faces right	2427606	—
DR 5000	2495402	Fill line faces user	2427606	—
DR 3900	2495402	Fill line faces user	2427606	LZV846 (A)
DR 3800, DR 2800, DR 2700	2495402	Fill line faces right	2122800	LZV584 (C)

Before starting the test:

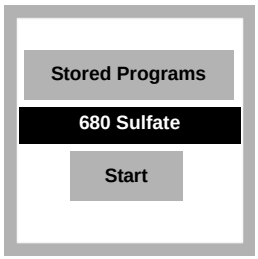
Adjust the standard curve for each new lot of reagent (*Standard solution method*).
For best results, calibrate the instrument with each new lot of reagent (see *Calibration*).
For best results, measure a reagent blank value for each new lot of reagent (follow the procedure using deionized water in place of the sample). Subtract the reagent blank value from the final results or enter the value as a reagent blank adjust for automatic subtraction.
Filter highly colored or turbid samples using filter paper and a funnel. Use this sample in step 2 and 5.
The Pour-Thru Cell cannot be used with this procedure.
SulfaVer® 4 contains barium chloride. The final solution will contain barium chloride (D005) at a concentration regulated as a hazardous waste by the Federal RCRA. Refer to a current MSDS for safe handling and disposal instructions.
Use a blank AccuVac® Ampule in place of the sample cell in Step 5 .if necessary.

Collect the following items:

Description	Quantity
Powder Pillow Test:	
SulfaVer® 4 Reagent Powder Pillows	1
Sample Cells (see *Instrument-specific information*)	2
AccuVac Test:	
SulfaVer® 4 Reagent AccuVac® Ampuls	1
Beaker, 50-mL	1
Sample Cells (see *Instrument-specific information*)	1
Stopper	1

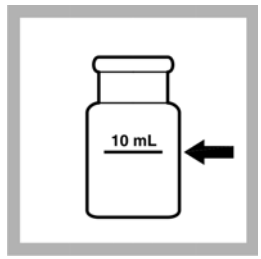
See *Consumables and replacement items* for reorder information.

SulfaVer 4 powder pillow procedure



1. Select the test.

Insert an adapter if required (see *Instrument-specific information*). Refer to the user manual for orientation.



2. **Prepared sample:** Fill a sample cell with 10 mL of sample.

3. Add the contents of one SulfaVer 4 Reagent Powder Pillow to the sample cell. Swirl vigorously to dissolve the powder.

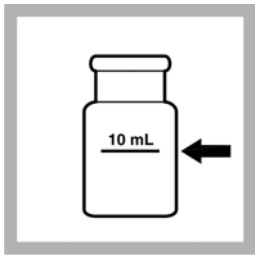
White turbidity will form if sulfate is present.

4. Start the instrument timer.

A five-minute reaction time will begin. Do not disturb the cell during this time.

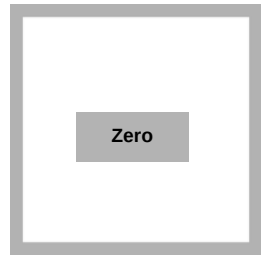
Note: *Accuracy is not affected by undissolved powder.*

SulfaVer 4 powder pillow procedure (continued)



5. Blank preparation: Fill a second sample cell with 10 mL of sample.

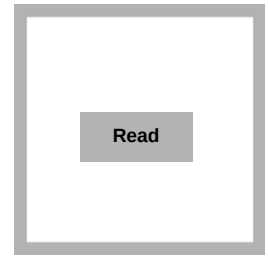
6. When the timer expires, wipe the blank and insert it in the cell holder (fill lines face right.)



7. ZERO the instrument.

The display will show:

0 mg/L SO_4^{2-}

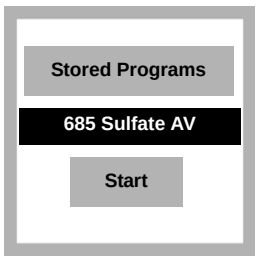


8. Within five minutes after the timer expires, wipe the cell and insert the prepared sample in the cell holder.

READ the results in mg/L SO_4^{2-}.

Clean sample cells with soap and a brush.

SulfaVer 4 AccuVac® Ampuls procedure



1. Select the test.

Insert an adapter if required (see *Instrument-specific information*). Refer to the user manual for orientation.

2. Prepared sample: Collect at least 40 mL of sample in a 50-mL beaker.

Fill a SulfaVer 4 Reagent AccuVac® Ampul with sample from the beaker. Keep the tip immersed while the Ampul fills completely.

3. Cap or stopper the Ampul and quickly invert several times to mix.

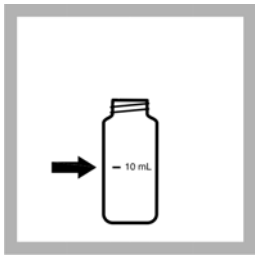
White turbidity will form if sulfate is present.

4. Start the instrument timer.

A five-minute reaction time will begin. Do not disturb the cell during this time.

Note: *Accuracy is not affected by undissolved powder.*

SulfaVer 4 AccuVac® Ampuls procedure (continued)



5. Blank Preparation: Fill a clean sample cell with 10 mL of sample.

6. When the timer expires, wipe the blank and insert it in the cell holder.

ZERO the instrument.

The display will show:

0 mg/L SO_4^{2-}

7. Within five minutes after the timer expires, wipe the Ampul and insert it in the cell holder.

READ the results in mg/L SO_4^{2-}.

Interferences

Table 389 Interfering substances

Interfering substance	Interference level
Calcium	Greater than 20,000 mg/L as $CaCO_3$
Chloride	Greater than 40,000 mg/L as Cl^-
Magnesium	Greater than 10,000 mg/L as $CaCO_3$
Silica	Greater than 500 mg/L as SiO_2

Sample collection, preservation and storage

Collect samples in clean plastic or glass bottles. Samples may be stored up to 7 days by cooling to 4 °C (39 °F) or lower. Warm to room temperature before analysis.

Accuracy check

Standard additions method (sample spike)
Required for accuracy check:

- Sulfate Ampule Standard Solution, 2500 mg/L sulfate
- Ampule breaker
- TenSette Pipet and pipet tips
- Mixing cylinder, 25 mL or 50 mL
- Beaker, 50 mL

1. After reading test results, leave the sample cell (unspiked sample) in the instrument.
2. Select standard additions from the instrument menu: **OPTIONS>MORE>STANDARD ADDITIONS**.

3. Accept the default values for standard concentration, sample volume and spike volumes. After the values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.
4. Open the standard solution ampule.
5. Fill three mixing cylinders with 25 mL of sample. Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL of standard, respectively, to each mixing cylinder and mix thoroughly. Transfer 10 mL of each sample spike to a clean sample cell.

   Note: *For AccuVac® Ampuls, fill three mixing cylinders with 50 mL of sample and spike with 0.2 mL, 0.4 mL and 0.6 mL of standard. Transfer 40 mL from each of the three mixing cylinders to three 50-mL beakers.*

6. Follow the *SulfaVer 4 powder pillow procedure* for each of the spiked samples, starting with the 0.1 mL sample spike. Measure each of the spiked samples in the instrument.

   Note: *For AccuVac Ampuls, follow the SulfaVer 4 AccuVac® Ampuls procedure for each of the spiked samples, starting with the 0.2 mL sample spike. Measure each of the spiked samples in the instrument.*

7. Select **GRAPH** to view the results. Select **IDEAL LINE** (or best-fit) to compare the standard addition results to the theoretical 100% recovery.

Standard solution method
Required for accuracy check:

- Sulfate standard solution, 1000 mg/L
- 100 mL Class A volumetric flask
- Tensette pipet, 1–10 mL and pipet tips

1. Prepare a 70 mg/L sulfate standard solution as follows: use a pipet to add 7.0 mL of sulfate standard solution, 1000 mg/L as SO_4^{2-}, to a 100-mL volumetric flask. Dilute to the mark with deionized water. Mix well. Prepare this solution daily.
2. Follow the *SulfaVer 4 powder pillow procedure* or *SulfaVer 4 AccuVac® Ampuls procedure* and use the 70-mg/L SO_4^{2-} standard solution in place of the sample.
3. To adjust the calibration curve using the reading obtained with the standard solution, set standard adjust to on (**OPTIONS**>(**MORE**)>**STANDARD ADJUST)** and accept the concentration.

Calibration

A calibration is recommended for the SulfaVer 4 method for the best accuracy. Complete the following steps to enter a new calibration curve in the instrument. Perform this procedure for each new lot of reagent.

Required items:

- Sulfate standard solution, 1000 mg/L
- Seven 100 mL Class A volumetric flasks
- 1–10 mL TenSette pipet and tips

1. Prepare seven calibration standards (10, 20, 30, 40, 50, 60 and 70 mg/L SO_4^{2-}) as follows. Use the Tensette pipet to add 1, 2, 3, 4, 5, 6 and 7 mL of the 1000-mg/L sulfate standard solution to seven different 100-mL Class A volumetric flasks.
2. Dilute each flask to the mark with deionized water. Mix thoroughly.
3. Use each standard solution in place of the sample and follow the *SulfaVer 4 powder pillow procedure* or *SulfaVer 4 AccuVac® Ampuls procedure*.

4. Refer to the user manual (user programs section) to enter the calibration in the instrument as a user program.

Method performance

Program	Standard	Precision 95% Confidence Limits of Distribution	Sensitivity Concentration change per 0.010 Abs change
680	40 mg/L SO_4^{2-}	30–50 mg/L SO_4^{2-}	0.4 mg/L SO_4^{2-}
685	40 mg/L SO_4^{2-}	32–48 mg/L SO_4^{2-}	0.7 mg/L SO_4^{2-}

Summary of method

Sulfate ions in the sample react with barium in the SulfaVer 4 and form a precipitate of barium sulfate. The amount of turbidity formed is proportional to the sulfate concentration. Test results are measured at 450 nm.

Consumables and replacement items

Required reagents

Description	Quantity/Test	Unit	Catalog number
SulfaVer® 4 Reagent Powder Pillows	1	100/pkg	2106769
OR			
SulfaVer® 4 Sulfate Reagent AccuVac® Ampuls	1	25/pkg	2509025

Required apparatus

Description	Quantity	Unit	Catalog number
AccuVac snapper	1	each	2405200
Beaker, 50-mL	1	each	50041H
Sample cell, 10 mL, round, 25 x 54 mm	1	each	2122800
Sample cell, 10 mL round, 25 x 60 mm	1	each	2427606
Sample cell, 10 mL, square, matched pair	2	2/pkg	24954025
Stopper	1	6/pkg	173106

Recommended standards

Description	Unit	Catalog number
Sulfate Standard Solution, 1000-mg/L	500 mL	2175749
Sulfate Standard Solution, 2500-mg/L, 10-mL Ampules	16/pkg	1425210
Mixed Parameter Standard for sulfate, fluoride, nitrate and phosphate	500 mL	2833049

Optional reagents and apparatus

Description	Unit	Catalog number
Cylinder, mixing, 25-mL	each	189640
Cylinder, mixing, 50-mL	each	189641
Blank AccuVac Ampules	25/pkg	2677825
Ampule Breaker, for 10 mL Ampules	each	2196800
Tensette Pipet 0.1–1.0 mL	each	1970001
Tips for Tensette Pipet 1–10	50/pkg	2185696
Tensette Pipet 1–10	each	1970010
Tips for tensette Pipet 1–10	50/pkg	2199796
Flask, volumetric, Class A, 100-mL	each	1457442

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Edition 7

Sulfide

DOC316.53.01136

USEPA[1] Methylene Blue Method[2] — Method 8131

(5 to 800 µg/L)

Scope and Application: For testing total sulfides, H_2S, HS^-, and certain metal sulfides in groundwater, wastewater, brines and seawater.

[1] USEPA approved for reporting wastewater analysis. Procedure is equivalent to Standard Method 4500-S^{2-} D.

[2] Adapted from *Standard Methods for the Examination of Water and Wastewater.*

Test preparation

How to use instrument-specific information

The *Instrument-specific information* table displays requirements that may vary between instruments. To use this table, select an instrument then read across to find the corresponding information required to perform this test.

Table 393 Instrument-specific information

Instrument	Sample volume	Sample cell	Cell orientation
DR 6000	10 mL	2495402	Fill line faces right
DR 5000	10 mL	2495402	Fill line faces user
DR 3900	10 mL	2495402	Fill line faces user
DR 3800, DR 2800, DR 2700	10 mL	2495402	Fill line faces right

Before starting the test:

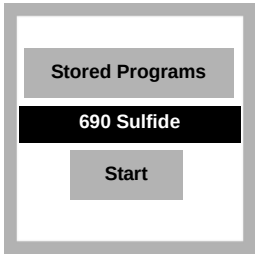
Analyze samples immediately. Do not preserve for later analysis.
Avoid excessive agitation of samples to minimize sulfide loss.
Some sulfide loss may occur if dilution is necessary.
Sulfide 2 reagent contains potassium dichromate. The final solution will contain hexavalent chromium (D007) at a concentration that is regulated as a hazardous waste by Federal RCRA. Refer to the current MSDS for safe handling and disposal instructions.

Collect the following items:

Description	Quantity
Sulfide 1 Reagent	1–2 mL
Sulfide 2 Reagent	1–2 mL
Water, deionized	10–25 mL
Pipet, serological, 10-mL	1
Pipet Filler, safety bulb	1
Sample Cells (see *Instrument-specific information*)	2
Stoppers	2

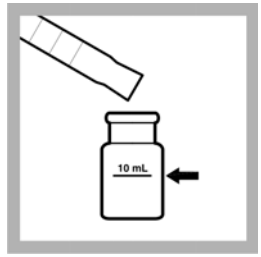
See *Consumables and replacement items* for reorder information.

Methylene Blue Method

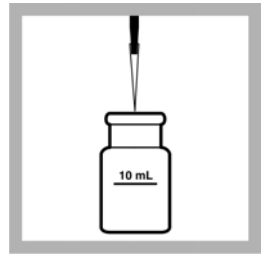


1. Select the test.

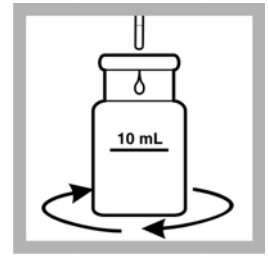
Insert an adapter if required (see *Instrument-specific information*). Refer to the user manual for orientation.



2. Blank Preparation: Measure 10 mL of deionized water in a sample cell.



3. Prepared Sample: Use a pipet to add 10 mL of sample to a second sample cell. Do not mix the sample more than necessary to prevent sulfide loss.



4. Use the dropper to add 0.5 mL Sulfide 1 Reagent to each cell.

Swirl to mix.

5. Use the dropper to add 0.5 mL Sulfide 2 Reagent to each cell.

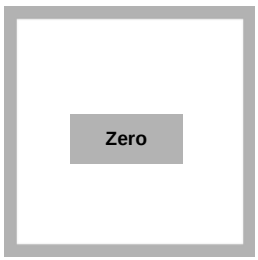
6. Cap or stopper the cell and immediately invert to mix.

The solution will turn pink initially and then turn blue if sulfide is present.

7. Start the instrument timer.

A five-minute reaction time will begin.

8. When the timer expires, wipe the blank and insert it in the cell holder.

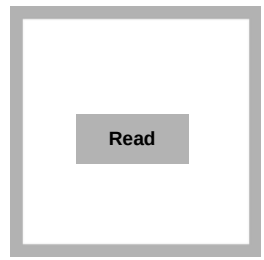


9. **ZERO** the instrument.

The display will show:

0.00 µg/L S^{2-}

10. Wipe the prepared sample and insert it in the cell holder.



11. **READ** the results in µg/L S^{2-}.

Soluble sulfides

Complete the following steps to measure soluble sulfides.

1. Centrifuge a sample in completely filled, capped tubes.
2. Use the supernatant in place of the sample and follow the *Methylene Blue Method* procedure.

To estimate insoluble sulfides, subtract the soluble sulfide concentration from the total sulfide concentration.

Interferences

Table 394 Interfering substances

Interfering substance	Interference level
Strong reducing substances such as sulfite, thiosulfate and hydrosulfite.	Interfere by reducing the blue color or preventing its development.
Sulfide, high levels	High concentrations of sulfide may inhibit full color development and require sample dilution. Some sulfide loss may occur when the sample is diluted.
Turbidity	For turbid samples, prepare a sulfide-free blank as follows. Use this blank in place of the deionized water blank in the *Methylene Blue Method* test procedure. 1. Measure 25 mL of sample into a 50-mL Erlenmeyer flask. 2. Add bromine water by drops with constant swirling until a permanent yellow color just appears. 3. Add phenol solution by drops until the yellow color just disappears. Use this solution to replace the deionized water in step 2 of the procedure. This pretreatment procedure removes sulfide from the sample, but the turbidity and any color will remain. The interference from turbidity or color will be corrected when the instrument is set to zero with this solution (step 9).

Sample collection, preservation and storage

Collect samples in clean plastic or glass bottles. Fill completely and cap tightly. Prevent excessive shaking or prolonged exposure to air. Analyze samples immediately.

Method performance

Program	Instrument	Standard	Precision 95% Confidence Limits of Distribution	Sensitivity Concentration change per 0.010 Abs change
690	DR 5000	520 µg/L S^{2-}	504–536 µg/L S^{2-}	5µg/L S^{2-}

Summary of method

Hydrogen sulfide and acid-soluble metal sulfides react with N,N-dimethyl-p-phenylenediamine sulfate to form methylene blue. The intensity of the blue color is proportional to the sulfide concentration. High sulfide levels in oil field waters may be determined after proper dilution. Test results are measured at 665 nm.

Consumables and replacement items

Required reagents

Description	Quantity/Test	Unit	Catalog number
Sulfide Reagent Set, includes:	—	—	2244500
Sulfide 1 Reagent	1 mL	100 mL MDB	181632
Sulfide 2 Reagent	1 mL	100 mL MDB	181732
Water, deionized	10 mL	4 liters	27256

Required apparatus

Description	Quantity	Unit	Catalog number
Pipet, serological, 10-mL	1	each	53238
Pipet Filler, safety bulb	1	each	1465100
Stopper, for 18-mm Tube	2	6/pkg	173106

Optional reagents and apparatus

Description	Unit	Catalog number
Bromine Water, 30 g/L	29 mL	221120
Phenol Solution, 30 g/L	29 mL	211220
Stopper, for 18-mm Tube	25/pkg	173125
Flask, Erlenmeyer, 50 mL	each	50541

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Iron, Ferrous

DOC316.53.01049

1-10 Phenanthroline Method[1]

Method 8146

0.02 to 3.00 mg/L

Powder Pillows or AccuVac® Ampuls

Scope and Application: For water, wastewater and seawater

[1] Adapted from *Standard Methods for the Examination of Water and Wastewater*, 15th ed. 201 (1980)

Test preparation

How to use instrument-specific information

The *Instrument-specific information* table displays requirements that may vary between instruments. To use this table, select an instrument then read across to find the corresponding information required to perform this test.

Table 195 Instrument-specific information

Instrument	Powder pillows		AccuVac Ampuls	
	Sample cell	Cell orientation	Sample cell	Adapter
DR 6000	2495402	Fill line faces right	2427606	—
DR 5000	2495402	Fill line faces user	2427606	—
DR 3900	2495402	Fill line faces user	2427606	LZV846 (A)
DR 3800, DR 2800, DR 2700	2495402	Fill line faces right	2122800	LZV584 (C)

Before starting the test:

For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water instead of the sample. Subtract the reagent blank value from the final results or perform a reagent blank adjust.
Analyze samples as soon as possible to prevent air oxidation of ferrous iron to ferric iron, which is not determined.
If ferrous iron is present, an orange color will form after adding the reagent.

Collect the following items:

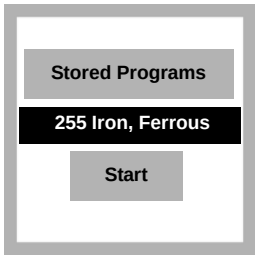
Description	Quantity
Powder Pillow Test:	
Ferrous Iron Reagent Powder Pillows	1
Sample Cells (see *Instrument-specific information*)	2
AccuVac Test:	
Ferrous Iron Reagent AccuVac® Ampuls	1
Beaker, 50 mL (AccuVac test)	1
Sample Cell (see *Instrument-specific information*)	1

Collect the following items:

Description	Quantity

See *Consumables and replacement items* for reorder information.

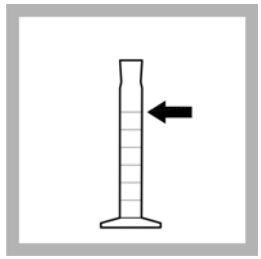
1-10 Phenanthroline method for powder pillows



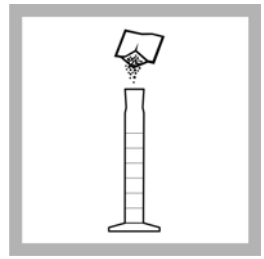
1. Select the test.

Insert an adapter if required (see *Instrument-specific information*).

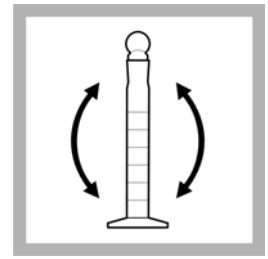
Refer to the user manual for orientation.



2. Fill a clean graduated mixing cylinder with 25 mL of sample.



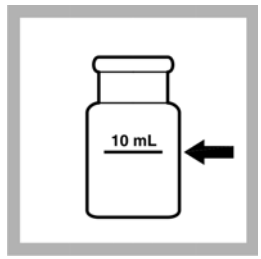
3. Prepared Sample: Add the contents of one Ferrous Iron Reagent powder pillow to the cylinder.



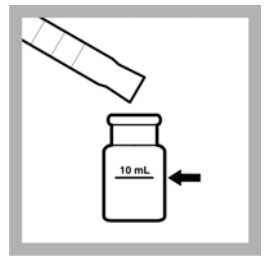
4. Insert a stopper and invert to mix. Undissolved powder does not affect accuracy.

5. Start the instrument timer.

A three-minute reaction period will begin.

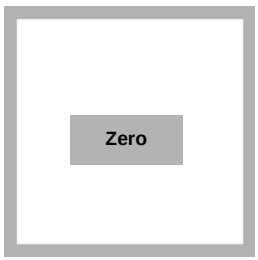


6. Blank Preparation: Fill a sample cell with 10 mL of sample.



7. Fill a second sample cell with the prepared sample from the mixing cylinder in step 4.

8. When the timer expires, insert the blank into the cell holder.

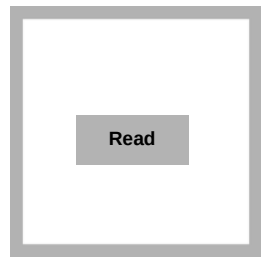


9. **ZERO** the instrument.

The display will show:

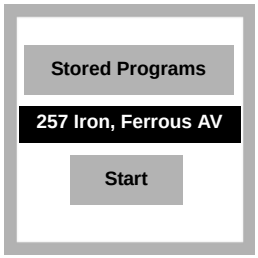
0.00 mg/L Fe^{2+}

10. Insert the prepared sample into the cell holder.



11. **READ** the results in mg/L Fe^{2+}.

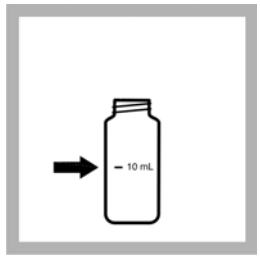
1-10 Phenanthroline method for AccuVac® Ampuls



1. Select the test.

Insert an adapter if required (see *Instrument-specific information*).

Refer to the user manual for orientation.



2. Blank Preparation: Fill a round sample cell with 10 mL of sample.

3. Prepared Sample: Fill a Ferrous Iron Reagent AccuVac® Ampul with sample from the beaker. Keep the tip immersed while the Ampul fills completely.

4. Quickly invert the Ampul several times to mix.

5. Start the instrument timer.

A three-minute reaction period will begin.

6. Wipe the blank and insert it into the cell holder.

ZERO the instrument.

The display will show:

0.00 mg/L Fe^{2+}

7. Wipe the Ampul and insert it into the cell holder.

READ the results in mg/L Fe^{2+}.

Sample collection, preservation and storage

- Collect samples in plastic or glass bottles.
- Analyze samples as soon as possible after collection.

Accuracy check

Standard solution method

Note: *Refer to the instrument user manual for specific software navigation instructions.*

Required for accuracy check:

- Ferrous Ammonium Sulfate, hexahydrate, 0.7022 g
- 1 L Class A volumetric flask
- 100 mL Class A volumetric flask
- Deionized water

- Analytical balance
- 2 mL Class A volumetric pipet and pipet filler

1. Prepare a 100 mg/L Fe^{2+} ferrous iron stock solution as follows:
   a. Dissolve 0.7022 grams of Ferrous Ammonium Sulfate, hexahydrate, in deionized water.
   b. Dilute to one liter in a Class A volumetric flask.
   c. In a 100 mL Class A volumetric flask, dilute 2.00 mL of this solution to 100 mL with deionized water to make a 2.0 mg/L standard solution. Prepare this solution immediately before use.
2. Follow the *1-10 Phenanthroline method for powder pillows* or the *1-10 Phenanthroline method for AccuVac® Ampuls* test procedure.
3. To adjust the calibration curve using the reading obtained with the 2.00-mg/L Standard Solution, select Options>More>Standard Adjust from the instrument menu.
4. Turn on the Standard Adjust feature and accept the displayed concentration. If an alternate concentration is used, enter the concentration and adjust the curve to that value.

Method performance

Program	Instrument	Standard	Precision 95% Confidence Limits of Distribution	Sensitivity Concentration change per 0.010 Abs change
255	DR 5000	2.00 mg/L Fe^{2+}	1.99–2.01 mg/L Fe^{2+}	0.021 mg/L Fe^{2+}
257	DR 2800	2.00 mg/L Fe^{2+}	1.98–2.02 mg/L Fe^{2+}	0.023 mg/L Fe^{2+}

Summary of method

The 1-10 phenanthroline indicator in the Ferrous Iron Reagent reacts with ferrous iron (Fe^{2+}) in the sample to form an orange color in proportion to the iron concentration. Ferric iron (Fe^{3+})does not react. The ferric iron concentration can be determined by subtracting the ferrous iron concentration from the results of a total iron test. Test results are measured at 510 nm.

Consumables and replacement items

Required reagents

Description	Quantity/Test	Unit	Catalog number
Ferrous Iron Reagent Powder Pillows	1	100/pkg	103769
OR			
Ferrous Iron Reagent AccuVac® Ampuls	1	25/pkg	2514025

Required apparatus

Description	Quantity/Test	Unit	Catalog number
Beaker, 50 mL	1	each	50041H
Sample cell, 10 mL round, 25 x 54 mm	1	each	2122800
Sample cell, 10 mL round, 25 x 60 mm	1	6/pkg	2427606
Sample cell, 10 mL square, matched pair	2	2/pkg	2495402

Recommended standards and apparatus

Description	Unit	Catalog number
Balance, analytical, 80 g x 0.1 mg 100–240 VAC	each	2936701
Ferrous Ammonium Sulfate, hexahydrate, ACS	113 g	1125614
Flask, volumetric, 1000 mL	each	1457453
Pipet filler, safety bulb	each	1465100
Pipet, volumetric, 2.00 mL	each	1451535
Water, deionized	4 L	27256
Wipers, disposable	280/pkg	2097000

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Edition 7

Iron, Total

DOC316.53.01053

USEPA[1] FerroVer® Method[2] **Method 8008**

0.02 to 3.00 mg/L **Powder Pillows or AccuVac® Ampuls**

Scope and Application: For water, wastewater and seawater; digestion is required for determining total iron

[1] USEPA approved for reporting wastewater analysis, *Federal Register,* June 27, 1980; 45 (126:43459)

[2] Adapted from *Standard Methods for the Examination of Water and Wastewater.*

Test preparation

How to use instrument-specific information

The *Instrument-specific information* table displays requirements that may vary between instruments. To use this table, select an instrument then read across to find the corresponding information required to perform this test.

Table 198 Instrument-specific information

Instrument	Powder pillows		AccuVac Ampuls	
	Sample cell	Cell orientation	Sample cell	Adapter
DR 6000	2495402	Fill line faces right	2427606	—
DR 5000	2495402	Fill line faces user	2427606	—
DR 3900	2495402	Fill line faces user	2427606	LZV846 (A)
DR 3800, DR 2800, DR 2700	2495402	Fill line faces right	2122800	LZV584 (C)

Before starting the test:

Digestion is required for determining total iron for EPA reporting purposes. Use the mild or vigorous digestion. Refer to the *Water Analysis Guide* for more information.
For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water in place of the sample. Subtract the reagent blank value from the final results or perform a reagent blank adjust. See the user manual for more information.
Adjust pH of stored samples before analysis.
For turbid samples, treat the blank with one 0.1-g scoop of RoVer Rust Remover. Swirl to mix.

Collect the following items:

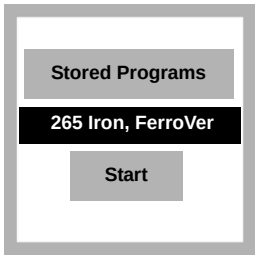
Description	Quantity
Powder Pillow Test:	
FerroVer® Iron Reagent Powder Pillow	1
Sample Cells (see *Instrument-specific information*)	2

Collect the following items: (continued)

Description	Quantity
AccuVac® Ampul test:	
FerroVer® Iron Reagent AccuVac® Ampul	1
Beaker, 50-mL	1
Sample Cells (see *Instrument-specific information*)	1
Stopper of 18 mm tubes	1

See *Consumables and replacement items* for reorder information.

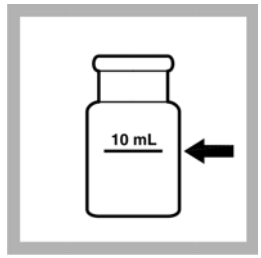
FerroVer method for powder pillows



1. Select the test.

Insert an adapter if required (see *Instrument-specific information*).

Refer to the user manual for orientation.



2. Prepared sample: Fill a clean sample cell with 10 mL of sample

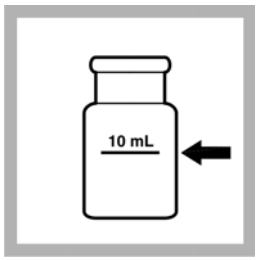
3. Add the contents of one FerroVer Iron Reagent Powder Pillow to the sample cell. Swirl to mix.

Accuracy is not affected by undissolved powder.

4. Start the instrument timer.

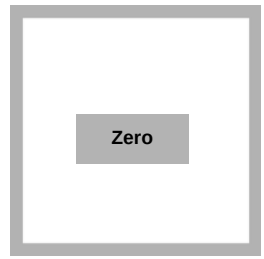
A three-minute reaction period will begin. An orange color will form, if iron is present.

(Allow samples that contain rust to react for at least 5 minutes.)



5. Blank preparation: Fill a second sample cell with 10 mL of sample.

6. When the timer expires, insert the blank into the cell holder.



7. ZERO the instrument.

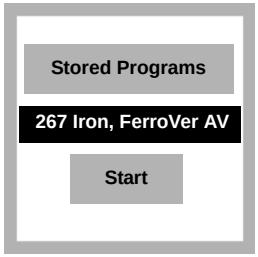
The display will show:

0.00 mg/L Fe

8. Insert the prepared sample into the cell holder.

READ the results in mg/L Fe.

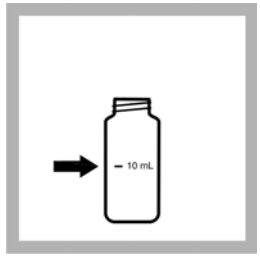
FerroVer method for AccuVac® Ampuls



1. Select the test.

Insert an adapter if required (see *Instrument-specific information*).

Refer to the user manual for orientation.



2. Blank Preparation: Fill a round sample cell with 10 mL of sample.

3. Prepared Sample: Collect at least 40 mL of sample in a 50-mL beaker. Fill a FerroVer Iron AccuVac® Ampul with sample from the beaker. Keep the tip immersed while the Ampul fills completely.

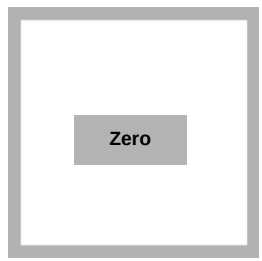
4. Quickly invert the Ampul several times to mix.

Accuracy is not affected by undissolved powder.

5. Start the instrument timer.

A three-minute reaction period will begin. An orange color will develop if iron is present.

6. Wipe the blank and insert it into the cell holder.



7. **ZERO** the instrument.

The display will show:

0.00 mg/L Fe

8. Wipe the Ampul and insert it into the cell holder.

READ the results in mg/L Fe.

Interferences

Table 199 Interfering substances

Interfering substance	Interference level
Calcium, Ca^{2+}	No effect at less than 10,000 mg/L as $CaCO_3$.
Chloride, Cl^-	No effect at less than 185,000 mg/L.
Copper, Cu^{2+}	No effect. Masking agent is contained in FerroVer Reagent.
High Iron Levels	Inhibit color development. Dilute sample and re-test to verify results.
Iron Oxide	Requires mild, vigorous or Digesdahl digestion. After digestion, adjust sample to pH 3–5 with sodium hydroxide, then analyze.
Magnesium	No effect at 100,000 mg/L as calcium carbonate.

Table 199 Interfering substances (continued)

Interfering substance	Interference level
Molybdate Molybdenum	No effect at 50 mg/L as Mo.
High Sulfide Levels, S^{2-}	1. Treat in fume hood or well-ventilated area. Add 5 mL hydrochloric acid[1], ACS to 100 mL sample in a 250 mL Erlenmeyer flask. Boil 20 minutes. 2. Cool. Adjust pH to 3–5 with Sodium Hydroxide[1]. Readjust volume to 100 mL with deionized water. 3. Analyze using *FerroVer method for powder pillows* or *FerroVer method for AccuVac® Ampuls*.
Turbidity	1. Add 0.1 g scoop of RoVer® Rust Remover to the blank. Swirl to mix. 2. Zero the instrument with this blank. 3. If sample remains turbid, add three 0.2 g scoops of RoVer to a 75 mL sample. Let stand 5 minutes. 4. Filter through a Glass Membrane Filter and Filter Holder[1]. 5. Use the filtered sample as the prepared sample and the blank.
Extreme Sample pH	Adjust pH to 3–5.
Highly Buffered Samples	Adjust pH to 3–5.

[1] See *Optional reagents and apparatus*.

Sample collection, preservation and storage

- Collect samples in acid-cleaned glass or plastic containers. No acid addition is necessary if analyzing the sample immediately.
- To preserve samples, adjust the pH to 2 or less with concentrated nitric acid (about 2 mL per liter). Preserved samples may be stored up to six months at room temperature.
- Before analysis, adjust the pH to between 3 and 5 with 5.0 N Sodium Hydroxide Standard Solution.
- Correct the test result for volume additions.
- If only dissolved iron is to be determined, filter the sample before acid addition.

Accuracy check

Standard additions method (sample spike)
Required for accuracy check:

- Iron Voluette® Ampule Standard, 25 mg/L
- Ampule breaker
- TenSette Pipet and Pipet Tips

1. After reading test results, leave the sample cell (unspiked sample) in the instrument.
2. Select Options>More>Standard Additions from the instrument menu.
3. Accept the default values for standard concentration, sample volume and spike volumes. After the values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.
4. Open the standard solution ampule.
5. Prepare a 0.1 mL sample spike by adding 0.1 mL of standard to 10 mL of unspiked sample. Start the instrument timer. After the timer expires, read the result.

6. Prepare a 0.2 mL sample spike by adding 0.1 mL of standard to the 0.1 mL sample spike. Start the instrument timer. After the timer expires, read the result.
7. Prepare a 0.3 mL sample spike by adding 0.1 mL of standard to the 0.2 mL sample spike. Start the instrument timer. After the timer expires, read the result.
8. Select **GRAPH** to view the results. Select **IDEAL LINE** (or best-fit) to compare the standard addition results to the theoretical 100% recovery.

Standard additions method for AccuVac Ampuls (sample spike)

1. Fill three mixing cylinders each with 50 mL of sample and spike with 0.2 mL, 0.4 mL and 0.6 mL of standard. Stopper and invert to mix.
2. Transfer 40 mL from each of the three mixing cylinders to three 50 mL beakers.
3. Analyze each standard addition sample as described in the *FerroVer method for AccuVac® Ampuls*.
4. Accept each standard additions reading. Each addition should reflect approximately 100% recovery.

Standard solution method

Note: *Refer to the instrument user manual for specific software navigation instructions.*

Required for accuracy check:

- Iron Standard Solution, 100 mg/L
- 100-mL volumetric flask
- Class A volumetric pipet, 2 mL
- Deionized water
- Pipet filler

1. Prepare a 2.00-mg/L Fe standard solution as follows:
   a. Pipet 2.00 mL of Iron Standard Solution, 100 mg/L, into a 100 mL volumetric flask.
   b. Dilute to the mark with deionized water. Mix well. Prepare this solution daily.
2. Use the 2.00 mg/L Fe standard solution in place of the sample. Follow the *FerroVer method for powder pillows* test procedure.
3. To adjust the calibration curve using the reading obtained with the Standard Solution, select Options>More>Standard Adjust from the instrument menu.
4. Turn on the Standard Adjust feature and accept the displayed concentration. If an alternate concentration is used, enter the concentration and adjust the curve to that value. Mixed-parameter standards are also available to simulate various matrices.

Method performance

Program	Standard	Precision 95% Confidence Limits of Distribution	Sensitivity Concentration change per 0.010 Abs change
265	2.00 mg/L Fe	1.99–2.01 mg/L Fe	0.021 mg/L Fe
267	2.00 mg/L Fe	1.98–2.02 mg/L Fe	0.023 mg/L Fe

Summary of method

FerroVer Iron Reagent converts all soluble iron and most insoluble forms of iron in the sample to soluble ferrous iron. The ferrous iron reacts with the 1-10 phenanthroline indicator in the reagent to form an orange color in proportion to the iron concentration. Test results are measured at 510 nm.

Consumables and replacement items

Required reagents

Description	Quantity/Test	Unit	Catalog number
FerroVer® Iron Reagent Powder Pillows (for 10-mL sample)	1	100/pkg	2105769
OR			
FerroVer® Iron Reagent AccuVac® Ampuls	1	25/pkg	2507025

Required apparatus

Description	Quantity	Unit	Catalog number
Beaker, 50 mL	1	each	50041H
Sample cell, 10 mL round, 25 x 54 mm	1	each	2122800
Sample cell, 10 mL round, 25 x 60 mm	1	6/pkg	2427606
Sample cell, 10 mL square, matched pair	2	2/pkg	2495402
Stopper for 18 mm tube	1	6/pkg	173106

Recommended standards

Description	Unit	Catalog number
Iron Standard Solution, 100 mg/L	100 mL	1417542
Iron Standard Solution, 10 mL Voluette® Ampule, 25 mg/L as Fe	16/pkg	1425310
Metals Drinking Water Standard, LR for Cu, Fe, Mn	500 mL	2833749
Metals Drinking Water Standard, HR for Cu, Fe, Mn	500 mL	2833649
Water, deionized	4 L	27256
Pipet, TenSette, 0.1–1.0 mL	each	1970001
Pipet Tips, for TenSette Pipet 1970001	50/pkg	2185696
Pipet Tips, for TenSette Pipet 1970001	1000/pkg	2185628
Flask, volumetric, Class A, 100 mL	each	1457442
Pipet, volumetric, Class A, 2.00 mL	each	1451536
Pipet Filler, safety bulb	each	1465100

Optional reagents and apparatus

Description	Unit	Catalog number
Beaker, 50 mL	each	50041H
Cylinder, mixing, 50 mL	each	189641
Hydrochloric Acid, concentrated	500 mL	13449
Nitric Acid, concentrated	500 mL	15249

Optional reagents and apparatus (continued)

Description	Unit	Catalog number
Sodium Hydroxide Standard Solution, 5.0 N	100 mL	245032
Glass Membrane Filter, 47 mm	100/pkg	253000
Glass Membrane Filter Holder	each	234000
RoVer Rust Remover	454 g	30001
Spoon, measuring, 0.1 g	each	51100

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Edition 7

Chloride

DOC316.53.01153

USEPA[1] Silver Nitrate Buret Titration Method[2]

Method 8225

0 to 25,000 mg/L as Cl^-

Buret Titration

Scope and Application: For water, wastewater and seawater.

[1] USEPA accepted for NPDES reporting when 0.0141 N silver nitrate standard solution is used.

[2] Adapted from *Standard Methods for the Examination of Water and Wastewater, (Standard Method 4500 Cl^- B).*

Test preparation

Before starting the test:

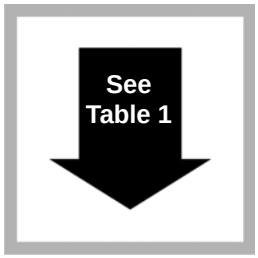
Adjust highly acidic or alkaline samples to a pH between pH 7 and 9. Use pH paper to measure the pH. A pH meter will contaminate the sample.
To calculate the result as mg/L sodium chloride (NaCl): mg/L chloride x 1.65 = mg/L sodium chloride
A small amount of silver nitrate is used to make the red-brown color in step 6. For most accurate results, follow the procedure using 100 mL of deionized water in place of the sample. Titrate this solution and note the volume of titrant required. For all samples, subtract this volume of titrant before calculating the mg/L chloride.

Collect the following items:

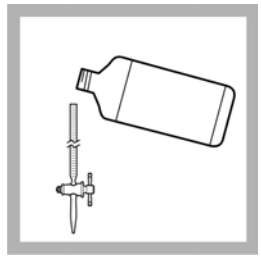
Description	Quantity
Chloride 2 Indicator Powder Pillow	1
Silver Nitrate Standard Solution (see *Range-specific information*)	1 bottle
Buret, Class A, 25-mL, with support stand	1
Erlenmeyer flask, 250 mL	1
Graduated cylinder	1

See *Consumables and replacement items* for reorder information.

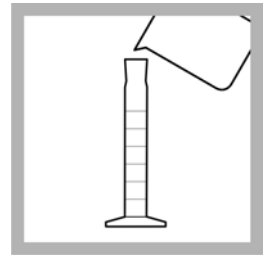
Buret titration



1. Select the sample volume and silver nitrate standard solution from *Range-specific information*.



2. Fill a 25-mL buret to the zero mark with the Silver Nitrate Standard Solution.



3. Use a graduated cylinder or pipet to measure the sample volume from *Range-specific information*.

4. Transfer the sample into a 250-mL Erlenmeyer flask. If the sample volume is less than 100 mL, dilute to approximately 100 mL with deionized water.

Buret titration (continued)

5. Add the contents of one Chloride 2 Indicator Powder Pillow. Swirl to mix.

6. Titrate the sample while swirling the flask until the color changes from yellow to red-brown.

7. Calculate:

mL titrant used x multiplier = mg/L chloride as Cl^-

Example: 100 mL of sample was titrated with the 0.0141 N silver nitrate solution and 15 mL of titrant was used to reach the endpoint.
The chloride concentration is: 15 x 5 = 75 mg/L as Cl^-

Table 87 Range-specific information

Range (mg/L as Cl^-)	Sample volume (mL)	Silver nitrate concentration	Multiplier
0–125	100	0.0141 N	5
100–250	50	0.0141 N	10
200–500	25	0.0141 N	20
500–1250	100	0.141 N	50
1000–2500	50	0.141 N	100
2500–10,000	25	0.141 N	200
5000–25,000	10	0.141 N	500

Interferences

An interfering substance can mask the end point. A dilution can reduce the interference to a level at which the substance does not interfere. If an interference is suspected, use a smaller amount of fresh sample and repeat the test.

Interfering substances lists substances that can interfere with this test.

Table 88 Interfering substances

Interfering substance	Interference level
Bromide	Cyanide, bromide and iodide interfere directly and are titrated as chloride.
Cyanide	Cyanide, bromide and iodide interfere directly and are titrated as chloride.
Iodide	Cyanide, bromide and iodide interfere directly and are titrated as chloride.
Iron	Iron concentrations over 20 mg/L will mask the end point.
Orthophosphate	Orthophosphate concentrations over 25 mg/L will cause a precipitate to form.
Sulfide	To remove interference from sulfide, add one Sulfide Inhibitor Reagent Powder Pillow to approximately 125 mL of the sample, mix for one minute and filter through filter paper.
Sulfite	To remove interference from at least 10 mg/L sulfite, add 3 drops of 30% hydrogen peroxide to 100 mL of sample before starting the test.

Sample collection, preservation and storage

Collect samples in clean plastic or glass bottles. Samples can be stored in sealed containers.

Accuracy check

Use the standard additions method to find if the sample has an interference. Use the standard solution method to make sure that the user has followed the test correctly and that the reagents are good.

Standard additions method (sample spike)
Required for accuracy check:

- Chloride Voluette® Ampule Standard, 12,500-mg/L as Cl^-
- Ampule breaker
- TenSette Pipet, 0.1–1.0 mL

Procedure for use with the **0.0141 N** titrant:

1. Open the standard solution ampule.
2. Use the TenSette Pipet to add 0.1 mL of the standard to the titrated sample. Swirl to mix.
3. Titrate the spiked sample to the end point. Write down the amount of titrant that was used to reach the end point.
4. Use the TenSette Pipet to add 0.2 mL of standard to the titrated sample. Swirl to mix.
5. Titrate the spiked sample to the end point. Write down the amount of titrant that was used to reach the end point.
6. Use the TenSette Pipet to add 0.3 mL of standard to the titrated sample. Swirl to mix.
7. Titrate the spiked sample to the end point. Write down the amount of titrant that was used to reach the end point.
8. Each 0.1 mL of standard that was added should use 2.5 mL of titrant to reach the endpoint. If more or less titrant was used, there can be an interference (see *Interferences*) or the concentration of the titrant has changed (see *Standard solution method*).

Procedure for use with the **0.141 N** titrant:

1. Open the standard solution ampule.
2. Use the TenSette Pipet to add 1.0 mL of the standard to the titrated sample. Swirl to mix.
3. Titrate the spiked sample to the end point. Write down the amount of titrant that was used to reach the end point.
4. Use the TenSette Pipet to add 2.0 mL of standard to the titrated sample. Swirl to mix.
5. Titrate the spiked sample to the end point. Write down the amount of titrant that was used to reach the end point.
6. Use the TenSette Pipet to add 3.0 mL of standard to the titrated sample. Swirl to mix.
7. Titrate the spiked sample to the end point. Write down the amount of titrant that was used to reach the end point.
8. Each 1.0 mL of standard that was added should use 2.5 mL of titrant to reach the endpoint. If more or less titrant was used, there can be an interference (see *Interferences*) or the concentration of the titrant has changed (see *Standard solution method*).

Standard solution method
A silver nitrate standard solution will slowly decompose with exposure to light. Complete the following test to make sure the concentration is accurate.

Required for accuracy check:

- Sodium Chloride Standard Solution, 1000-mg/L as Cl^-
- 100-mL Class A volumetric flask (for use with 0.0141 N titrant only)

Procedure for use with the **0.0141 N** titrant:

1. Add 10.0 mL of the sodium chloride standard solution, 1000-mg/L as Cl^-, to a 100-mL Class A volumetric flask. Dilute to 100 mL with deionized water and mix fully. This solution has a concentration of 100 mg/L chloride.
1. Add 100.0 mL of the diluted sodium chloride standard solution, 100-mg/L as Cl^-, to an Erlenmeyer flask.
2. Add the Chloride 2 indicator and swirl to mix.
3. Titrate the standard to the end point with the 0.0141 N silver nitrate titrant and calculate the result. If the result is more than 105 mg/L chloride, discard the silver nitrate titrant and replace it with a fresh supply.

Procedure for use with the **0.141 N** titrant:

1. Add 100.0 mL of the sodium chloride standard solution, 1000-mg/L as Cl^-, to an Erlenmeyer flask.
2. Add the Chloride 2 indicator and swirl to mix.
3. Titrate the standard to the end point with the 0.141 N silver nitrate titrant and calculate the result. If the result is more than 1050 mg/L chloride, discard the silver nitrate titrant and replace it with a fresh supply.

Summary of method

Silver nitrate is used as the titrant and potassium chromate as the indicator. Silver nitrate first reacts selectively with the chloride in the sample to produce insoluble white silver chloride. After all the chloride has been precipitated, the silver nitrate reacts with the chromate to form an orange or red-brown silver chromate precipitate.

Consumables and replacement items

Required reagents

Description	Quantity/Test	Unit	Catalog number
Chloride 2 Indicator Powder Pillows	1 pillow	50/pkg	105766
Titrant—select one or more based on range:			
Silver Nitrate Standard Solution, 0.0141 N	varies	1 L	31653
Silver Nitrate Standard Solution, 0.141 N	varies	500 mL	1255149

Required apparatus

Description	Quantity/Test	Unit	Catalog number
Buret, Class A, 25-mL	1	each	2636540
Buret Clamp, double	1	each	32800
Flask, Erlenmeyer, graduated, 250-mL	1	each	50546
Graduated cylinder—select one or more based on range:			
Cylinder, graduated, 10-mL	1	each	50838
Cylinder, graduated, 25-mL	1	each	50840
Cylinder, graduated, 50-mL	1	each	50841
Cylinder, graduated, 100-mL	1	each	50842
Support Stand	1	each	56300

Recommended standards

Description	Unit	Catalog number
Chloride Standard Solution, Voluette® Ampule, 12,500-mg/L as Cl^-, 10-mL	16/pkg	1425010
Sodium Chloride Standard Solution, 1000-mg/L as Cl^-	500 mL	18349
Voluette Ampule breaker, 10 mL	each	2196800

Optional reagents and apparatus

Description	Unit	Catalog number
Filter Paper, 12.5 cm diameter	100/pkg	69257
Hydrogen Peroxide, 30%	473 mL	14411
Sulfide Inhibitor Reagent Powder Pillow	100/pkg	241899
TenSette Pipet, 0.1 to 1.0 mL	each	1970001
Water, deionized	500 mL	27249
Pipet, TenSette®, Pipet, 1.0–10.0 mL	each	1970010
Pipet Tips, for TenSette Pipet 1970010[1]	50/pkg	2199796
Pipet Tips, for TenSette Pipet 1970010[1]	250/pkg	2199725
Pipet, TenSette, Pipet, 0.1–1.0 mL	each	1970001
Pipet Tips, for TenSette Pipet 1970001[1]	50/pkg	2185696
Pipet Tips, for TenSette Pipet 1970001[1]	1000/pkg	2185628
pH Paper, 0–14 pH range	100/pkg	2601300
Sampling Bottle with cap, low density polyethylene, 250 mL	12/pkg	2087076
Flask, Class A volumetric, 100 mL	each	1457442
Dropper, glass	5/pkg	1419705
Clippers for powder pillows	each	66800

[1] Other sizes are available

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Edition 7

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ESS Laboratory
Division of Thielsch Engineering
Cranston, RI

SOP NO. 20.0002A
MEASUREMENT OF PURGEABLE ORGANIC COMPOUNDS IN WATER BY CAPILLARY COLUMN GAS CHROMATOGRAPHY/MASS SPECTROMETRY (EPA METHOD 524.2)

REVIEWED BY:

Operations Manager	Date 11/17/6
QA Manager	Date 11/20/06
Laboratory Director	Date 11/20/06

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MEASUREMENT OF PURGEABLE ORGANIC COMPOUNDS IN WATER BY CAPILLARY COLUMN GAS CHROMATOGRAPHY/MASS SPECTROMETRY (EPA METHOD 524.2)

1.0 SCOPE AND APPLICATION

1.1 This method describes the technique utilized by ESS Labs in the qualitative and quantitative analysis of volatile samples based on EPA method 524.2. The following are compounds that ESS Laboratory currently determines by this method:

Analyte	Chemical Abstract Service Registry Number
Acetone	67-64-1
Benzene	71-43-2
Bromobenzene	108-86-1
Bromochloromethane	74-97-5
Bromodichloromethane	75-27-4
Bromoform	75-25-2
Bromomethane	74-83-9
2-Butanone	78-93-3
n-Butylbenzene	104-51-8
sec-Butylbenzene	135-98-8
tert-Butylbenzene	98-06-6
Carbon Disulfide	75-15-0
Carbon tetrachloride	56-23-5
Chlorobenzene	108-90-7
Chloroethane	75-00-3
Chloroform	67-66-3
Chloromethane	74-87-3
2-Chlorotoluene	95-49-8
4-Chlorotoluene	106-43-4
Dibromochloromethane	124-48-1
1,2-Dibromo-3-chloropropane	96-12-8
1,2-Dibromoethane	106-93-4
Dibromomethane	74-95-3
1,2-Dichlorobenzene	95-50-1
1,3-Dichlorobenzene	541-73-1
1,4-Dichlorobenzene	106-46-7
Dichlorodifluoromethane	75-71-8
1,1-Dichloroethane	75-34-3
1,2-Dichloroethane	107-06-2
1,1-Dichloroethene	75-35-4
cis-1,2-Dichloroethene	156-59-2
trans-1,2-Dichloroethene	156-60-5

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Analyte	Chemical Abstract Service Registry Number
1,2-Dichloropropane	78-87-5
1,3-Dichloropropane	142-28-9
2,2-Dichloropropane	590-20-7
1,1-Dichloropropene	563-58-6
cis-1,3-Dichloropropene	10061-01-5
trans-1,3-Dichloropropene	10061-02-6
Ethylbenzene	100-41-4
Hexachlorobutadiene	87-68-3
2-Hexanone	591-78-6
Isopropylbenzene	98-82-8
4-Isopropyltoluene	99-87-6
Methylene chloride	75-09-2
4-Methyl-2-Pentanone	108-10-1
Methyl-t-butyl ether	1634-04-4
Naphthalene	91-20-3
n-Propylbenzene	103-65-1
Styrene	100-42-5
1,1,1,2-Tetrachloroethane	630-20-6
1,1,2,2-Tetrachloroethane	79-34-5
Tetrachloroethene	127-18-4
Tetrahydrofuran	109-99-9
Toluene	108-88-3
1,2,3-Trichlorobenzene	87-61-6
1,2,4-Trichlorobenzene	120-82-1
1,3,5-Trichlorobenzene	108-70-3
1,1,1-Trichloroethane	71-55-6
1,1,2-Trichloroethane	79-00-5
Trichloroethene	79-01-6
Trichlorofluoromethane	75-69-4
1,2,3-Trichloropropane	96-18-4
1,2,4-Trimethylbenzene	95-63-6
1,3,5-Trimethylbenzene	108-67-8
Vinyl chloride	75-01-4
o-Xylene	95-47-6
m-Xylene	108-38-3
p-Xylene	106-24-3

1.2 This method is used to determine volatile organic compounds in drinking water samples.

1.3 This method can be used to quantify most volatile organic compounds that have boiling points below 200 °C and that are insoluble or slightly soluble in water. Volatile water-soluble compounds can be included in this analytical technique, however, for the more soluble compounds quantitation limits are approximately ten times higher because of poor purging efficiency.

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1.4 The quantitation limit of this method for an individual compound is approximately 0.5 µg/L for drinking water. Method Reporting Limits (MRL) will be proportionately higher for samples that require dilution or reduced sample size to avoid off-scale peaks.

2.0 METHOD SUMMARY

2.1 This SOP provides gas chromatograph/mass spectrometer (GC/MS) conditions for the detection of volatile organic compounds (VOCs).

2.2 This SOP only addresses sample analysis by purge and trap.

2.2.1 Purged sample components are trapped in a tube containing suitable sorbent materials.

2.2.2 When purging is complete, the sorbent tube is heated and back-flushed with helium to desorb components. The analytes are desorbed directly to a bore capillary column for analysis.

2.2.3 Wide bore capillary columns require a jet separator while narrow bore columns can be directly interfaced to the ion source.

2.3 A temperature program is used in the gas chromatograph to separate the organic compounds. Detection is achieved using a mass spectrometer (MS).

2.4 Tentative identifications are obtained by analyzing standards under the same conditions used for samples and comparing resultant GC Retention times. Absolute identifications are obtained by comparing the mass spectra of individual compounds to the reference spectra for that compound.

2.5 Concentrations of the identified compounds are measured by relating the response produced for the compound to the response produced by a compound that is used as an internal standard.

3.0 HEALTH AND SAFETY

3.1 Many chemicals used in this procedure are tentatively classified as known or suspected carcinogens. Extreme caution should be used in handling all chemicals in this procedure.

3.2 To minimize exposure, process samples in an exhaust hood or well-ventilated workspace. When working with samples and chemicals, wear gloves to minimize contact and possible absorption. Always wear appropriate eye protection.

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3.3 The laboratory employee, prior to attempting this procedure, should review proper emergency response to spills or injury. This includes location of spill kits, emergency eyewash and showers, fire fighting equipment, as well as evacuation routes.

3.4 Material Safety Data Sheets are available for all chemicals used in this procedure. All laboratory employees are required to read these before handling these chemicals.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

4.1 Non-chlorinated drinking water samples are preserved with 1:1 HCl at a ratio of 100 µL to 40 ml. All samples are collected in triplicate.

4.2 When sampling from an open body of water, such as surface water, wastewater and possible leachate samples, partially fill a 1 L beaker with sample from a representative area. Fill a sample vial with sample from the larger container and adjust the pH of the sample to about 2 by adding 1:1 HCl dropwise while stirring. Check the pH with narrow range pH paper and record the number of drops necessary to adjust the pH to 2.when collecting the actual samples, refill the beaker with fresh sample and pour thesample into sample vials following the filling instructions described in Attachments B or C. Add the appropriate number of drops of 1:1 HCl to each sample to adjust the pH to about 2. If samples are suspected to contain residual chlorine, add ascorbic acid or sodium thiosulfate as described in Attachments B or C.

4.3 When sampling from a faucet, flush for about 10 minutes before collecting the sample.

4.4 When samples are suspected to contain residual chlorine, as is the case for samples received from treatment facilities, 25 mg of ascorbic acid per 40 ml sample is added to the vials before they are sent to the client (samples are unlikely to have residual chlorine present in excess of 5 mg/L). After the vials are filled with sample, two drops of 1:1 HCl are added to each vial. Sampling instructions are provided in Attachments B and C. ***NOTE: Samples to be analyzed only for Trihalomethanes are only preserved with Sodium thiosulfate.***

4.5 Samples for analysis using this SOP must be stored in tightly sealed vials with Teflon-lined silicon septum seals in which the original sample was collected. All samples are chilled when collected and stored in a refrigerator at 4 ± 2 °C within the laboratory.

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4.6 Samples for volatile analysis must be analyzed within 14 days of sample collection.

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4.7 The trip blanks are prepared with organic free water and sample preservatives. This water comes from a carbon filtration system located in the VOA lab. A trip blank must accompany each batch of VOC samples at all times.

4.8 Refrigerator blanks are prepared like trip blanks and stored in the refrigerator with samples. These blanks are analyzed weekly to check for possible cross contamination from samples. To prevent cross contamination from sample to sample, all sample containers must be airtight and all high level samples must be stored separately. If the blank is greater than the MRL, then notify the operations manager immediately.

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

5.1 During analysis, major contaminant sources are volatile materials in the laboratory and impurities in the inert purging gas and in the sorbent trap. The use of Teflon tubing, Teflon thread sealant or flow controllers with rubber components in the purging device should be avoided since such materials out-gas organic compounds which will be contracted in the trap during the purge operation. Analyses of laboratory reagent blanks provide information about the presence of contaminants. When potential interfering peaks are noted in laboratory reagent blanks, the analyst should change the purge gas source and regenerate the molecular sieve purge gas filter. Subtracting blank values from sample results in not permitted.

5.2 Interfering contamination may occur when a sample containing low concentrations of volatile organic compounds is analyzed immediately after a sample containing relatively high concentrations of volatile organic compounds. A preventive technique is between sample rinsing of the purging apparatus and sample syringes with two portions of reagent water. After analysis of a sample containing high concentrations of volatile organic compounds, one or more laboratory reagents blanks should be analyzed to check for cross contamination.

5.3 Special precautions must be taken to determine methylene chloride. The analytical and sample storage area should be isolated from all atmospheric sources of methylene chloride, otherwise random background levels will result. Since methylene chloride will permeate Teflon tubing, all GC carrier gas lines and purge gas plumbing should be constructed of stainless steel or copper tubing. Laboratory worker's clothing should be cleaned frequently since clothing previously exposed to methylene chloride fumes during common liquid/liquid extraction procedures can contribute to sample contamination.

5.4 Traces of ketones, methylene chloride and some other organic solvents can be present even in the highest purity methanol. This is another potential source of contamination, and should be assessed before standards are prepared in the methanol.

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5.5 This procedure can be used to quantitate most volatile organic compounds that have boiling points below 200 °C and that are insoluble or slightly soluble in water. Volatile water-soluble compounds can be included in this analytical technique. However, for the more soluble compounds, quantitation limits are approximately ten times higher because of poor purging efficiency. Such compounds include low-molecular weight halogenated hydrogenated hydrocarbons, aromatics, ketones, nitriles, acetates, acrylates, ethers, and sulfides.

5.6 Meta- and para- xylenes are coeluters. They are evaluated and reported as a combined result.

5.7 On the VRX column, 2-chlorotoluene and n-propylbenzene are not completely resolved and they share the same primary ion. The shoulder is integrated based on the secondary ion. These analytes are routinely evaluated and manually integrated when necessary.

6.0 EQUIPMENT/APPARATUS

6.1 Microsyringes: 10 µL, 25 µL, 50 µL, 100 µL, 250 µL, 500 µL, and 1000 µL. These syringes should be equipped with a 20 gauge (0.006 ″ ID) needle.

6.2 Disposable micro-pipettes: 10, 50, 100, and 200 µL volumes. Purchased from Drummond.

6.3 Micro-reaction vessels: 1.0 ml and 5.0 ml purchased from Supelco, catalog. No 3-3293 with Mininert caps. Clean reaction vessels by placing in oven at 105 °C overnight.

6.4 Volumetric flasks: of various sizes with ground glass stoppers.

6.5 Vials: 40 ml, with pierceable Teflon screwcap top.

6.6 Disposable Pasteur pipettes.

6.7 Purge and Trap Devices: (A) Aquatek 70 Liquid Auto-sampler. (B) Teledyne Tekmar Purge and Trap Concentrator.

6.7.1 Aquatek autosampler: The Aquatek Purge and Trap Autosampler is designed to automate the tedious sample handling procedures associated with purge and trap analyses for volatile organic compounds (VOCs) under current EPA methods. The Aquatek can be used for drinking water and waste water.

6.7.1.1 The Aquatek can be programmed using Teklink software to run up to 70 samples. All method parameters are pre-set by the analyst. The Aquatek automatically takes a 25 ml aliquot directly

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from the VOA vial, injects 2µL of Internal Standard/surrogate standard, and transfers this to a 25 ml sparge tube to be purged by the Stratum Purge and Trap Concentrator.

6.7.2 The purge gas passes through the water column as finely divided bubbles, each with a diameter of less than 3 mm at the origin. Purge flow of 40ml/min for 11 min.

6.7.3 The trap is at least 25 cm long and has an inside diameter of at least 0.105 ". This trap is commercially available from Teledyne Tekmar (Purge & Trap #9), Catalog No. 14-9908-403.

6.7.4 The desorber is capable of rapidly heating to 250°C. Desorb preheat at 245°C. Desorb Temp 250°C. Desorb time of 4 min.

6.8 Gas Chromatograph: HP-6890. A complete analytical system with a temperature programmable gas chromatograph suitable for on-column injection and all required accessories, including syringes, analytical columns, and gases.

6.9 Column: 20m x 0.18mm ID available from Agilent (DB-VRX). Recommended by Agilent for optimized performance on a 5973 MSD.

6.10 Mass spectrometer HP 5973MSD. Capable of scanning from 35-200 amu every second or less utilizing 70 volts electron energy in the electron impact ionization mode producing mass spectra that meets all the method criteria when 25 ng 4-Bromofluorobenzene (BFB) is injected through the gas chromatography inlet.

6.11 Data system: HP MS Chemstation. This system is interfaced to the mass spectrometer detector and allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer software allows searching of any GC/MS data files for ions of a specified mass and plotting such ion abundance versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). The software is also capable of integrating the abundance of any EICP between specified time or scan number limits. The most current version of the EPA/NIST Mass Spectral Library is also available.

7.0 REAGENTS AND STANDARDS

7.1 Reagents

7.1.1 **Methanol**: Purge and Trap Grade, purchased from Fisher (catalog no. A453-500).

7.1.2 **Reagent Water**: Water where interferent is not observed at the Method Detection Limit (MDL) of the parameters of interests. Water is purified using the Nanopure ultra-pure water system located in the VOA lab.

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7.1.3 **Hydrochloric acid** (1+1): Carefully add measured volume of concentrated HCl to equal volume of reagent water.

7.1.4 **Sodium Thiosulfate** – 10% Solution is prepared and 2 drops are added to vials that are sent out to treatment facilities.

7.1.5 **Ascorbic Acid** - Crystalline Powder, U.S.P.-F.C.C. Grade, J.T. Baker

7.2 **Standards**

7.2.1 **Primary Standards**: obtained from a commercial source and stored in the freezer at –10 °C to –20 °C. After opening store in a vial with a mininert valve. All certificates of analysis are marked with the Primary standard ID and placed in a logbook. Primary standards are not to be used after the manufacturer's expiration date and once opened; it may not be used after 4 weeks. Methanol solutions prepared from gaseous analytes are not stable for more than 1 week when stored at <0C. At room temperature they must be discarded after one day. A fresh gas working standard is prepared weekly. The gases are evaluated for stablility by comparing the CCV (primary std) to the Blank Spike control sample (Second source std) daily. Inconsistancies (> 30%) require a new Gas ampule. The following primary standards are used:

Primary Standard	Manufacturer	Cat. #	Concentration
Ketones	Restek	30006	5000 µg/ml
Cal Mix #1	Restek	30633	2000 µg/ml
Custom VOA Additions	Restek	558360	2000 µg/ml
Oxygenates	Restek	30465	2000 µg/ml /10,000 µg (TBA)
Gases	Restek	30042-510	2000 µg/ml
Vinyl Acetate	Restek	30216	2000 µg/ml
1,4-Dioxane*	Supelco	44-2251	Neat
1,3,5-Trichlorobenzene	Accustandard	AS-E0176	5000 µg/ml

*Prepare 1,4-Dioxane working standard by adding 10 µl into 1 ml of methanol; final concentration is 10,000 µg/ml

7.2.2 Working Calibration- In a 5 ml volumetric flask, add 62.5 µL Gases, 125 µL – Ketones, 62.5 µL – Cal mix #1, 62.5 µL – Custom VOA additions, 25 µL 1,3,5-Trichlorobenzene 62.5 µL Oxygenates, 237.5 µl of 1,4-Dioxane, 62.5 µL Vinyl Acetate. Volumize to 5ml with methanol. Final concentration will be 125 µg/ml Ketones and tert-Butyl Alcohol; 500 µg/ml 1,4-Dioxane and 25 µg/ml all other compounds.

7.2.3 Secondary Source Standards – obtained from a commercial source.

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Secondary Standard	Manufacturer	Cat. #	Concentration
Custom standard	Ultra	CUS-10604	2000 µg/ml
2-CEVE standard	Accustandard	M-601C-10x	2000 µg/ml
VOC Mix	Ultra	DWM-592	2000 µg/ml
Vinyl Acetate	Accustandard	App-9-211-	2000 µg/ml
502.2 Mix	Accustandard	M-502-10x	2000 µg/ml
Ketone Mix	Accustandard	Clp-022k-25X	2000 µg/ml
1,4-Dioxane*	Ultra	RCC-180	Neat
tert-Butyl Alcohol	Accustandard	S-410	2000 µg/ml

*Prepare 1,4-Dioxane working standard by adding 10 µL into1 ml of methanol. Final concentration is 10,000 µg/ml.

7.2.3.1 Initial Calibration verification /Laboratory Control /Matrix Spike standard(s) (ICV/LCS/MS) used to check the primary standard. Into a 5 ml volumetric flask add 62.5 µL each of Custom standard, 2-CEVE standard, VOC Mix #2, Vinyl acetate standard, and 502.2 Mix. Add 100 µL of Ketone mix, 312.5 µL tert-Butyl Alcohol and 250 µL of 1,4-Dioxane working standard. Volumize to 5 ml with methanol. Final concentration will be 125 µg/L for Ketones and tert-Butyl Alcohol, 500 µg/L for 1,4-Dioxane, and 25 µg/L for all other compounds.

7.2.4 Surrogate standards – The surrogate solution is made up of 4-Bromofluorobenzene and 1,2-dichlorobenzene-d4. The mixed stock surrogate is purchased from Supelco at 2000µg/ml catalog no. 4-8466. The stock solution is logged into the primary standard logbook upon receipt.

7.2.5 The Internal Standard is Fluorobenzene and is purchased from Restek at a concentration of 2000 µg/ml catalog no. 30030.

7.2.5.1 Surrogate/Internal Standard Mix prepared by adding 312.5 µL of the stock surrogate and internal standards to a 10 ml volumetric flask. Volumize to 10 ml with methanol, final concentration 62.5 µg/ml. Transfer to an Aquatek standard vial and put vial into the Aquatek standard well. The Aquatek injects 2µL into each 25 ml sample, final concentration 5 µg/ml.

7.2.6 4-Bromofluorobenzene (BFB) standard –purchased from Supelco at a concentration of 2000 µg/ml catalog no. 4-8083.

7.2.6.1 Add 62.5 µL of stock solution to a 5.0 ml volumetric flask. Volumize with methanol, final concentration 25 µg/ml. 1 µL is injected directly into the column to check the instrument's tune.

7.2.7 Matrix Spiking Standard – See section 7.2.3.1.

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7.2.7.1 Working matrix spike solution – Addition of 40 µL to 100 ml of sample yields a concentration of 50 µg/L for Ketones and 10 µg/L for all other target compound.

8.0 PROCEDURE

8.1 Initial calibration for purge-and-trap procedures:

8.1.1 GC/MS operating conditions: The following parameters are setup through Hewlett Packard MS EnviroQuant. A copy of a EnviroQuant method is in Attachment A.

8.1.2

Electron energy:	70 volts
Mass range:	35 amu - 260 amu
Scan time:	3.25 sec/scan
Initial column temperature:	45°C
Initial column holding time:	3 min.
Column temperature program:	36°C/min(Rate1), 20°C/min(Rate 2).
Final column temperature:	190°C/min(Rate1), 225°C/min (Rate 2).
Final column holding time:	2.0 min.
Injector temperature:	150°C
Transfer line temperature:	150°C
Carrier gas(Helium):	37.1 ml/min.

8.1.3 Assemble a purge-and-trap device that meets the specification in Section 6.7. New traps are conditioned one hour at 270°C in the bake mode.

8.1.4 Prior to any analysis of samples or standards, the run logbook must be filled out. Each instrument has its own run logbook. Any unusual observations are included in the comment section of the logbook.

8.1.5 A tune check must be performed before the initial calibration curve. Inject 1µl (25 ng) of the 4-Bromofluorobenzene standard (See section 7.2.6) onto the GC/MS column. Analysis is not to begin until the 4-BFB spectra meets the criteria in Section 11.1. The analysis of all calibration standards must be accomplished within 12 hours of this tune check.

8.1.6 Prepare the standards for generating the calibration curve using the Working Standards (see section 7.2.2) and Internal Standard/Surrogate solution (see Section 7.2.5.1) as follows:

8.1.6.1 To 50 ml of reagent water in a volumetric flask, inject 20 µl of the Working standard for a final concentration of 10 µg/L. Cap, invert three times, and transfer to VOA vial leaving no headspace. Seal tightly and place in Aquatek auto-sampler for analysis. Repeat this procedure six times using 0.2, 1.0, 4.0, 8.0,

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40, 80 and 160 µL for concentrations of 0.1, 0.5, 2, 4, 20, 40, and 80 µg/L, respectively, to complete the calibration curve.

8.1.7 Carry out the purge-and-trap analysis procedure as described in Section 8.3.

8.1.8 EnviroQuant will tabulate the area response of the characteristic ions (see Table 1) against concentration for each compound and internal standard. It will also calculate response factors (RF) for each compound relative to its internal standards according to the calculations in Section 9.1.

8.1.8.1 NOTE: It is each analyst's responsibility to become familiar with the software by reading the available manuals. **After generating the initial calibration curve in Enviroquant, the analyst must visually check that each calibration standard was entered into the new calibration method. This is accomplished by checking that the area response for one compound from each calibration standard's printout corresponds to the area account listed in the calibration method in Enviroquant.** Each analyte along with their retention times and the ions used for qualitative analysis are listed in Appendix B.

8.1.9 The average RF is calculated in the EnviroQuant Method for each compound.

8.1.10 Using the RFs from the initial calibration, EnviroQuant software calculates the percent relative standard deviation (%RSD) for all compounds using the equation in Section 9.2. The %RSD should be less than 20% for each compound.

8.1.10.1 Linearity - If the %RSD of any compound is 20% or less, then the relative response factor is assumed to be constant over the calibration range, and the average relative response factor may be used for quantitation.

8.1.10.2 When the RSD exceeds 20%, the plotting and/or visual inspection of the calibration data can be a useful diagnostic tool. The inspection may indicate analytical problems, including errors in standard preparation, the presence of active sites in the chromatographic system, analytes that exhibit poor chromatographic behavior, etc.

8.1.10.3 If a %RSD greater than 30 percent is measured for any method analyte, then corrective action to eliminate a system leak and/or column reactive sites is required before re-attempting calibration. Poor purging compounds are listed in Table 1.

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8.1.9.4 If the RSD of ANY compound exceeds 20% (never to exceed 30%), then one of the following options must be applied to the GC/MS initial calibration in this situation or a new initial calibration must be performed.

8.1.9.4.1 Adjust the instrument and/or perform instrument maintenance until the RSD of the calibration meets the 20% QC limit. This option would apply in those instances where a linear instrument response is expected.

8.1.9.4.2 Narrow the calibration range until the response is linear. If the low standard is below the estimated quantitation limit (i.e., for the poor purgers in a commercially available prepared standard mix), then this standard may be dropped. Recalculate the RSD without this standard to see if the RSD meets the QC limit. It would be recommended that a new standard be prepared at a concentration between the existing fourth and fifth calibration standards, analyzed, and a new RSD calculated with all five points.

8.1.9.4.3 Use a linear calibration. This would be achieved by performing a linear regression of the instrument response versus the concentration of the standards. In order to be used for quantitative purposes, the coefficient of determination (R^2) must be greater than or equal to 0.99. This can easily be accomplished within the EnviroQuant software.

8.1.9.4.4 Use a quadratic regression or non-linear calibration model, minimum of six points for this option. This option should be used only after exhausting the other three options, or in situations where the analyst knows that the instrument response does not follow a linear model over a sufficiently wide working range. (Not an option for South Carolina samples)

8.2 Daily GC/MS analysis: Just prior to all samples and standard analysis, the run logs are filled out according to 8.1.3.

8.2.1 Prior to the analysis of samples, inject 1 µL of the 4-Bromofluorobenzene standard (Section 7.2.6). The resultant mass spectra for BFB must meet all of the criteria in Section 11.1 before sample analysis begins. This criteria must be met each 12-hour shift.

8.2.2 The initial calibration curve must be verified each 12-hour shift. This is accomplished by analyzing a calibration standard at 10 µg/L. The

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acceptance criterion is ± 30% deviation or drift, for poor purging compounds (Ketones, 2-CEVE), 50%. Compounds not analyzed for in the batch of samples do not need to meet the criteria.

8.2.3 The internal standard responses and retention times in the continuing calibration standard should be evaluated immediately after data acquisition. If the area for any of the internal standards changes by ± 30% (or – 50% to +100% from the initial calibration) or the retention time changes by ±0.5 minutes from the last daily calibration standard check, the mass spectrometer should be checked for defects. Analysis is not to continue until problem has been resolved.

8.2.4 Method blank analysis should be performed at the following frequency:

8.2.4.1 For the analysis of volatile compounds, a method blank analysis must be performed once for each 12-hour shift and must be analyzed before any samples.

8.2.4.2 A method blank for volatile analysis should contain less than or equal to the MRL of methylene chloride or 5 ppb in the case of 25 ml purge.

8.2.4.3 For all other TCL compounds not listed above, the method blank should be:

8.2.4.3.1 Less than ½ the MRL or less than the level of acceptable blank contamination specified in the approved quality assurance project plan.

8.2.4.3.2 Less than 5% of the regulatory limit associated with an analyte.

8.2.4.3.3 Or less than 5% of the sample result for the same analyte, whichever is greater.

8.2.4.4 If a laboratory method blank exceeds these criteria, the operator should consider the analytical system to be out of control. The source of the contamination should be investigated and appropriate corrective measures should be taken and documented before further sample analysis proceeds. All samples processed with a method blank that is out of control (i.e., contaminated) should be re-extracted/re-purged and reanalyzed. Any deviation will be noted on the customer's report. See section 11.0 for expanded criteria.

8.3 GC/MS analysis: All samples and standards are analyzed under the same conditions in the associated initial calibration. Run logs are filled according to 8.1.3.

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8.3.1 All samples and standard solutions should be allowed to warm to ambient temperature before analysis.

8.3.2 Set up the GC/MS system as outlined in Section 8.1.1.

8.3.3 BFB tuning criteria and daily GC/MS calibration criteria must be met (Section 11.1) before analyzing samples.

8.3.4 Place sample in sealed VOA vial in Aquatek auto-sampler for analysis.

8.3.5 The Aquatek will automatically inject 2µL of surrogate/internal standard QC mix (Section 7.2.5.1) to the sample. The surrogate/internal standard QC mix is to be added to each continuing calibration standard, blank, sample and spiked sample.

8.3.6 Purge the sample for 11.0 ±0.1 min at ambient temperature, then dry purge for 2 minutes.

8.3.7 At the conclusion of the purge time, the purge-and-trap will sound a ready signal from the GC and begin to desorb the trap while initiating the chromatographic temperature program and the CHEMSTATION data acquisition.

8.3.8 The Aquatek auto-sampler will automatically rinse the purge chamber with reagent water.

8.3.9 After the sample has been desorbed for 1 minute, the LSC 3000 will recondition the trap by heating to 260°C while back flushing with Helium. This step will take 10 minutes

8.3.10 When necessary to composite samples for analysis, the following procedure is used:

8.3.10.1 Add equal amounts of sample to a 50 ml volumetric flask. Pour off composited sample to a VOA vial leaving no headspace and seal tightly.

8.3.10.2 The samples must be cooled to 4°C or less during this step to minimize volatilization losses. Sample vials may be placed in a tray of ice during the processing.

8.3.10.3 Place composited VOA vial in Aquatek autosampler for analysis.

8.3.11 If a sample or a dilution of a sample has a concentration of analytes that exceeds the initial calibration range, the sample must be re-analyzed at a higher dilution. All dilutions must be performed manually in a 50 ml volumetric flask using reagent water and an appropriate amount of sample.

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8.3.11.1 When a sample is analyzed that has any target compound at a level exceeding twice the initial calibration range, this analysis should be followed by a blank reagent water analysis in a different purge chamber to demonstrate that system is clean. If the blank analysis is not free of interferences, the system should be decontaminated. Sample analysis may not resume until a blank can be analyzed that is free of interferences. Analysis of sample in the contaminated chamber may not resume until the analysis of reagent water in the chamber is free of any target compounds.

NOTE: Since the analytical system is automated the analyst may not be aware that a high concentration sample has been analyzed. If analysis of subsequent samples occurs, then the presence of the high concentration analyte makes reanalysis of that sample necessary to determine if carryover has occurred.

8.3.12 For matrix spike and matrix spike duplicate analysis, add 40 µL of the matrix spike solution (Section 7.2.3.1) to 100 ml of sample. Disregarding any dilution, this is equivalent to a concentration of 10 µg/L of each matrix spike compound.

8.3.13 All dilutions should keep the response of the major constituents in the upper half of the linear range of the curve.

8.4 Data interpretation

8.4.1 Qualitative identification

8.4.1.1 An analyte is identified by comparison of the sample mass spectrum with the mass spectrum of a standard of the compound obtained on the user's GC/MS. These standard reference spectra are obtained through analysis of the calibration standards. To compare the sample and reference spectra, go into the EnviroQuant software (QEDIT). The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity - any ions over 30% if less than three such ions are present.

8.4.1.2 The requirements for qualitative verification by comparison of mass spectra are:

8.4.1.2.1 The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a

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compound-specific retention time will be accepted as meeting this criterion.

8.4.1.2.2 The RRT of the sample component is within ±0.06 RRT units of the RRT of the standard component.

8.4.1.2.3 The relative intensities of the characteristic ions agree within 20% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 30% and 70%).

8.4.1.2.4 Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs. ESS Laboratory currently reports total xylenes due to inability of resolving the isomers.

8.4.1.2.5 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important. Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra, and in qualitative identification of compounds. When analytes coelute (i.e., only one chromatographic peak is apparent), the identification criteria can be met, but each analyte spectrum will contain extraneous ions contributed by the coeluting compound.

8.4.1.2.6 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the type of analyses being conducted. When serving the role of QA (or referee) laboratory, tentatively identified compounds (TICs) shall always be reported. Guidelines for making tentative identification are:

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8.4.1.2.6.1 Relative intensities of major ions in the reference spectrum (ions >10% of the most abundant ion) should be present in the sample spectrum.

8.4.1.2.6.2 The relative intensities of the major ions should agree within ±30% to be consistent with target compound list identification. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 20 and 80%).

8.4.1.2.6.3 Molecular ions present in the reference spectrum should be present in the sample spectrum.

8.4.1.2.6.4 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

8.4.1.2.6.5 Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.

8.4.2 Quantitative Analysis

8.4.2.1 Compounds which have been identified are quantified by the internal standard method, utilizing the integrated abundance of the primary ion. The internal standard used shall be those listed in Section 8.1.7. The EnviroQuant method is setup to calculate the concentration of the analytes of interest in ng/ml using the initial calibration results stored in the method. This raw data number does not account for deviations in sample volume, sample weight, dilution factors, and % moisture. To include these other factors, see section 9.0.

9.0 CALCULATIONS

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9.1 Response Factor (RF)
RF=(As x Cis)/(Ais x Cs)

Where:

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A_S = Peak area (or height) of the analyte or surrogate.
A_{IS} = Peak area (or height) of the internal standard.
C_S = Concentration of the analyte or surrogate, in µg/L.
C_{IS} = Concentration of the internal standard, in µg/L.

9.2 Percent Relative Standard Deviation (%RSD)
% RSD = (SD/ $RF_{Average}$) x 100%

Where:
RSD = Relative standard deviation.
RF = Mean of 5 initial RFs for a compound.
SD = Standard deviation of average RFs for a compound.

9.3 Percent Difference
%Difference = (($RF_{Average}$ – RFc)/ RF Average) x 100%

Where:
$RF_{Average}$ = Average response factor from initial calibration
RF_C = Response factor from current verification check standard.

9.4 Percent Drift
%Drift = ((C1-C0)/ C1) x 100%

Where:
C_1 = compound standard concentration
C_0 = measured concentration using selected quantitation method

9.5 Concentration of Target Analytes in Water and Water-Miscible Waste
Concentration:
ug/L = (Ax)(Is)(D) / (Ais)($RF_{Average}$)(Vs)

Where:
A_X, A_{IS} = same as in section 9.1.
I_S = Mass (amount) of the internal standard in the concentrated sample extract (ng). This is not just the mass injected into the instrument, but the total mass of internal standard in the concentrated extract.
D = Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution was made, D = 1. The dilution factor is always unitless.
$RF_{Average}$ = Mean response factor from the initial calibration.
V_S = Volume of the aqueous sample extracted or purged (ml). If units of liters are used for this term, then multiply the results by 1000.

Using the units specified here for these terms will result in a concentration in units of ng/ml, which is equivalent to µg/L.

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NOTE: The result on the raw data generated through Enviroquant is the final result in ug/L without any dilution factor applied.

9.6 Concentration of Target Analytes in Sediment/Soil, Sludge, and Waste

10.0 QUALITY ASSURANCE/QUALITY CONTROL

10.1 **Accuracy and Precision**

All laboratory personnel must demonstrate initial proficiency for each sample preparation method/matrix that he or she performs. All new employees must successfully demonstrate initial proficiency prior to independently performing analysis on real samples. This must be accomplished by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The initial proficiency results will become part of each employee's training file

QC Sample Preparation:

Spiking Solution: Four QC samples must be prepared from a spiking solution with the analytes of interest. The spiking solution must be made using standards **prepared independently from those used for calibration**. The samples must be prepared at a concentration that would result in data falling within the middle of the calibration curve. In most cases the blank spike or matrix spike solution is used. Prep: The samples are prepared in a clean matrix. In most cases this initial demonstration will simply be a matter of preparing four blank spikes with a batch of samples.

Sample Analysis:

The four QC samples must be analyzed within the criteria of the method being evaluated. The QC samples must be handled in exactly the same manner as actual samples.

Accuracy Calculation:

Accuracy is defined as the closeness of agreement between an observed value and an accepted reference value. Each of the four spiked samples will be calculated for percent recovery. The average of the percent recovery values is the accuracy result.

Precision Calculation:

Precision is defined as the agreement of a set of replicate measurements without assumption of knowledge of the true value. Precision is estimated by the relative standard deviation (RSD) of the four QC samples.

$$\%RSD = (s / \bar{x})\ 100\ \%$$

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Where :

s = Standard Deviation of a finite number of values. On a scientific calculator use the **σxn-1** key.

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x = The average of the four QC sample % recoveries.

Reporting Accuracy and Precision

Accuracy and Precision data should be presented with the following minimuminfo:

Matrix: Prep Method:
Clean-up Method: Analysis Method:
Date Extracted: Date Analyzed:
Sample Prepared by: If Applicable Sample Analyzed by:
Accuracy: Precision:

Parameter	*% Rec. QC 1*	*% Rec. QC 2*	*% Rec. QC 3*	*% Rec. QC 4*	*Average Recovery*	*Standard Deviation*	*%RSD*

Interpretation of Results:

Method 524.2 has established accuracy precision ranges of 80-120% for all analytes and ≤ 20% RSD. If any of the accuracy and precision results do not fall within the criteria then re-prep and reanalyze all QC samples only for those analytes that were no good.

10.2 A preparation batch of samples is defined as a group of up to twenty field samples of similar matrix type that have been prepared at the same time or time sequence with the same lots of reagents for the same analysis. In addition to the twenty samples, each preparatory batch will contain at a minimum, a method blank, a laboratory control sample, a matrix spike, and a matrix spike duplicate. An analytical or instrumental batch is defined as samples that are analyzed together within the same time period or in continuous sequential time periods. Within the analytical batch are included individual QC requirements as defined by the analytical (determinative) method. For instance, each injection sequence would begin with a tune and CCV (or initial 7-point calibration and ICV), followed by a method blank, and then the other QC samples (normally a LCS, MS, MSD, etc.) and remaining field samples. Preparation batches of samples may be continuously strung together in these run sequences, as long as the analytical batch QC requirements meet the acceptance criteria established within the appropriate SOP. If all field and QC samples could not be completed within the 12-hour clock, then another tune, CCV, and method blank would be required. Each analytical sequence must be documented using the run log.

10.3 Perform BFB tune every 12 hours. Tuning acceptance criteria are presented in Section 11.1. The computer software will evaluate the tune information. The analyst should be aware of the process used. The criteria specified in Section 11.1 would typically be derived using three scans, one at the apex, and one on both sides of the apex.

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10.4 Run a 7-point initial calibration curve, using the primary source standards each time major instrument maintenance occurs, or if the CCV does not meet acceptance criteria. Acceptance criteria are presented in Section 11.2.

10.5 Run an initial calibration verification (ICV) standard using secondary standards (7.2.3) following the initial calibration curve. Acceptance criteria are listed in Section 11.3.

10.6 Run a mid-point Continuing Calibration Verification (CCV) at 10 µg/L using the primary source standards on a daily basis before sample analysis. Also run a CCV every 12 hours during an analytical sequence. Acceptance criteria are listed in Section 11.4.Also run a daily 1.0µg/L LCV (Low Level CCV) before sample analysis using the primary source standards. Trihalomethanes must meet 50 – 200% recovery criteria.

10.7 A method blank must be prepared with each batch of samples not to exceed 12 hours. The method blank should be prepared from organic-free water. The method blanks are to be related to each 12-hour sequence of samples injected. Acceptance criteria for these blanks are listed in Section 11.5. Instrument blanks may be injected at any time in the sequence to verify absence of contamination.

10.8 A laboratory control sample must be prepared and analyzed with each batch of samples. The LCS would be prepared using the second source standard and would contain all method target analytes. Control charts will be maintained bi-annually for the LCS for all target analytes and surrogate spikes. Surrogate spike recoveries from the method blanks will also be control charted along with the LCS surrogate values. Acceptance criterion is 70-130% (poor purgers are 50-150%).

NOTE: The CCV and LCS are considered to be equivalent. Only one will need to be analyzed. In the case of solids, the LCS is extracted similar to the samples.

10.9 On an ongoing basis the laboratory analyzes matrix spikes and matrix spike duplicates from each batch of 20 samples.

10.9.1 The matrix spike is prepared as described in Section 7.2.3.4.

10.9.2 Matrix spike recovery limits are calculated for each compound and each matrix. The recoveries limits are 70-130% with %RPD of $\leq$ 20%.

10.10 Surrogates recoveries are calculated for each LCS, method blank, QC sample, and field sample analyzed. Acceptance criteria are listed in Section 11.7.

10.11 The relative retention times (RRTs) of identified compounds need to be checked for each identified compound in samples, and compared to standard RRT. Acceptance criteria are presented in Section 11.8.

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10.12 Ion abundance for target compounds and any tentatively identified compounds need to meet specific requirements. Acceptance criteria for ion abundance are presented in Section 11.9.

10.13 Internal standard area counts for standards and samples must meet specifications as described in Section 11.10.

10.14 Data shall be checked to ascertain if it conforms to accepted practices. All sample analytical results used for final data reporting must be between the low standard and the high standard. Results, which fall below the low standard or above the high standard, are to be reported as estimated values. Corrective actions are described in Section 11.0.

10.15 MDLs are determined in either reagent water or organic-free sand / soil and verified annually. (Project-specific requirements may require that the MDL study be performed in the site-specific matrix.). MDLs (LODs) are determined in accordance with SOP 110.0013

11.0 DATA VALIDATION

All items shall be verified and documented using the data review checklist in Figure 4.

11.1 Ensure that the BFB tune was run at the beginning of each 12-hour sequence for each batch of samples analyzed. The acceptance criteria are listed below:

Table 1. BFB Key Ions and Ion Abundance Criteria

Mass	Ion Abundance Criteria
50	15.0-40.0% of mass 95
75	30.0-80.0% of mass 95
95	base peak, 100% relative abundance
96	5.0-9.0% of mass 95
173	< 2.0% if mass 174
174	> 50.0% of mass 95
175	5.0-9.0% of mass 174
176	> 95.0%, but less than 101% of mass 174
177	5.0-9.0% of mass 176

11.1.1 If the BFB acceptance criteria are not met, perform any or all of the following corrective actions:
Re-inject BFB.
Retune with PFTBA, then re-inject BFB.
Clean MS source, retune with PFTBA, re-inject BFB.

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11.1.2 If the tuning criteria still cannot be met after performing the above, have the mass spectrometer serviced by manufacturer representative.

11.2 After a seven-point initial calibration curve is analyzed, ensure that the following criteria were met.

11.2.1 For the RFs, the %RSD must be less than 30% for all method target analytes, except for the poor purgers where the %RSD must be less than 50%. If acceptance criteria are not met, the following corrective actions should be performed:

11.2.1.1 If the %RSD of any method target analyte is 20%, or less, then the relative response factor is assumed to be constant over the calibration range, and the average relative response factor may be used for quantitation.

11.2.1.2 If the %RSD of any method target analyte is greater than 20%, calibration curves must be constructed using first or higher order regression fits of the seven calibration points (five points may be used if linear regression is used). The corresponding coefficient of determination must be 0.99 or greater.

11.2.1.3 If these acceptance criteria are not met, then the following corrective actions should be performed: (1) adjust the instrument and/or perform instrument maintenance; or (2) narrow the calibration range using six standards at different concentrations. The low end of the calibration curves must be carefully watched.

11.3 If a 7-point initial calibration was performed, verify that an initial calibration verification (ICV) was performed. The percent difference for all analytes must be within 30%. If not, reanalyze the ICV or prepare a new calibration curve as necessary.

11.4 After the continuing calibration verification (CCV) standard is analyzed, ensure it was run at the required frequency (every 12 hours or initially before daily analysis). All analytes must be within ± 30% difference or drift(poor purgers ±50%). If criterion is not met, reanalyze the CCV or prepare a new calibration curve as necessary.

11.5 Assess the method blanks. The analyst shall confirm that this blank was analyzed at the required frequency and the criteria in Section 8.2.4 are met. Analytical batches with Method blanks outside acceptance criteria will be re-prepped and re-analyzed with the following exceptions:

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11.5.1 Samples that are that are at least twenty times higher than the method blank may be reported.

11.5.2 When the method blank is less than 5% of the regulatory limit associated with the analyte the method blank would be acceptable.

11.5.3 If the analyte is found in the method blank above the ½ MRL but is not in any of the associated samples, no corrective action is needed.

11.5.4 Any results that are reported with method blank contamination must be B-flagged.

11.6 Assess that matrix spike/matrix spike duplicates were analyzed at required frequency. Acceptance criteria are that all % Recovery and/or RPD results meet project-established goals. If no project goals are specified, then results must be within the indicated control limits on the appropriate blank spike control charts. If these conditions are not met, perform the following corrective actions as appropriate.

11.6.1 If both LCS and MS/MSD recoveries are unacceptable, then the entire batch of field and QC samples must be reanalyzed.

11.6.2 If the MS/MSD is unacceptable, but the LCS is acceptable, then a potential matrix effect has been identified. Reasonable attempts must be made to address matrix interference. Report matrix interference in the project narrative.

11.7 Check the surrogate calculations for correctness for all samples, blanks, LCS, MS, and MSD. It is recommended that the maximum default range be set to 80 to 120%, for Method 524. If blank or LCS are outside criteria, then stop analysis until problem has been corrected. If samples are outside criteria, re-analyze sample once. If still outside criteria, then note exception in the project narrative. If re-analysis produces results within criteria, then report out valid results only.

11.8 The relative retention times must be checked for all identified compounds in both standards and samples. The internal standard absolute retention times must also be checked for all analyses. Acceptance criteria are as follows:

11.8.1 The relative retention times of each compound in each calibration run should agree within 0.06 relative retention time (RRT) units. Late-eluting compounds usually have much better agreement.

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11.8.2 If the retention time for any internal standard changes by more than 30 seconds from the last daily calibration, the chromatographic system must be inspected for malfunctions and corrections must be made, as required.

11.9 The analyst must verify that ion abundance meet specific criteria for the various analyses. The following acceptance criteria shall be checked for all appropriate samples.

11.9.1 All ions present in the standard mass spectrum at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) must be present in the sample spectrum.

11.9.2 The relative intensities of ions specified in Section 11.9.1 must agree within plus or minus 20% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample abundance must be between 30 and 70 percent.)

11.9.2.1.1 Molecular ions present in the reference spectrum should be present in the sample spectrum.

11.9.2.1.2 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

11.9.2.1.3 Ions present in the reference spectrum, but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.

11.9.3 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. When serving the role as QA (or referee) laboratory, tentatively identified compounds (TICs) are always reported. Computer-generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches will the mass spectral interpretation specialist assign a tentative identification. Guidelines for making tentative identification are:

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11.9.3.1.1 Relative intensities of major ions in the reference spectrum (ions > 10% of the most abundant ion) should be present in the sample spectrum.

11.9.3.1.2 The relative intensities of the major ions should agree within ±30%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 20 and 80%.)

11.9.3.1.3 Molecular ions present in the reference spectrum should be present in the sample spectrum. Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

11.9.3.1.4 Ions present in the reference spectrum, but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.

11.10 The analyst will check the internal standard area counts for all calibration standards, QC samples, and samples for quantitation. If the area for any of the internal standards changes by 70 to 130% from the last daily calibration standard check or 50-200% of the initial calibration. The mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. In the event the internal standard area counts fail these criteria, the following corrective actions should be considered.

11.10.1 Check to be sure there are no errors in internal standards preparation or addition. Also check instrument performance.

11.10.2 Re-analyze the sample if any of the above checks do not reveal a problem.

11.11 The analyst must verify all reported results are derived from analytical results that are below the highest standard of the initial calibration curve and above the low standard. Values reported below the low standard are to be reported as estimated values (J values). For samples that exceed the calibration curve, dilute and analyze an appropriate sample aliquot.

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11.12 All manual integration must be printed when made for verification.

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11.13 Besides the items listed in Sections 11.1 through 11.12, the analyst should also verify the additional items as noted in Figure 4.

12.0 REFERENCES

12.1 Methods for the Determination of Organic Compounds in Drinking Water—Supplement III, EPA-600/R-95-131. August 1995

13.0 POLLUTION PREVENTION and WASTE MANAGEMENT

13.1 ESS Laboratory's policies on pollution prevention and waste management are covered in SOP 90_0002, Hazardous Waste Contingency and Emergency Response Plan. All employees are trained in the requirements of the SOP.

14.0 METHOD PERFORMANCE

14.1 Precision and Accuracy data must be generated by all employees before performing this analysis on client samples. The data is generated by analyzing a method blank and four blank spike samples. Acceptance criteria are 80-120% Recovery and %RSD of ≤ 20%. Five (5) sporadic marginal exceedances are allowed.

14.2 The precision and accuracy data in Table 1 were obtained using the SOP. Values are in ug/L.

15.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

Table 1. Typical Precision and Accuracy data generated 1/12/2006

Compound Name	Spk Amt	Avg	RSD	%Rec	Compound Name	Spk Amt	Avg	RSD	%Rec
Dichlorodifluoromethane	10	11.718	7	117	1,1,2-Trichloroethane	10	10.188	3	102
Chloromethane	10	11.390	3	114	1,3-Dichloropropane	10	10.305	2	103
Vinyl Chloride	10	12.420	4	124	Tetrachloroethene	10	9.060	3	91
Bromomethane	10	11.943	3	119	2-Hexanone	50	49.243	3	98
Chloroethane	10	11.548	4	115	Dibromochloromethane	10	10.115	3	101
Trichlorofluoromethane	10	11.965	4	120	1,2-Dibromoethane	10	10.063	2	101
Acetone	50	54.395	5	109	Chlorobenzene	10	10.203	2	102
Methyl Iodide	10	11.918	5	119	1,1,1,2-Tetrachloroethane	10	9.975	2	100
1,1-Dichloroethene	10	11.815	4	118	Ethylbenzene	10	10.418	3	104
Carbon Disulfide	10	11.363	3	114	Xylene P,M	20	20.538	3	103
Methylene Chloride	10	11.733	2	117	Xylene O	10	10.325	3	103
Acrylonitrile	10	11.170	1	112	Styrene	10	10.453	2	105
Methyl tert-Butyl Ether	10	9.815	1	98	Bromoform	10	9.803	3	98

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trans-1,2-Dichloroethene	10	10.600	4	106	Isopropylbenzene	10	10.290	2	103
1,1-Dichloroethane	10	10.593	2	106	1,2,3-Trichloropropane	10	9.885	3	99
cis-1,2 Dichloroethene	10	10.523	3	105	Bromobenzene	10	10.045	3	100
2,2-Dichloropropane	10	8.895	5	89	1,1,2,2-Tetrachloroethane	10	10.050	2	
2-Butanone	50	51.740	5	103	n-Propylbenzene	10	10.320	6	103
Bromochloromethane	10	10.935	2	109	2-Chlorotoluene	10	9.715	10	97
Tetrahydrofuran	10	9.123	4	91	4-Chlorotoluene	10	9.978	3	100
Chloroform	10	10.528	2	105	1,3,5-Trimethylbenzene	10	10.315	3	
1,1,1-Trichloroethane	10	9.810	3	98	tert-Butylbenzene	10	10.253	3	103
1,1-Dichloropropene	10	10.340	3	103	1,2,4-Trimethylbenzene	10	10.090	3	101
Carbon Tetrachloride	10	10.308	4	103	sec-Butylbenzene	10	10.345	4	103
Benzene	10	10.118	1	101	1,3 Dichlorobenzene	10	10.195	2	102
1,2-Dichloroethane	10	10.800	2	108	4-Isopropyltoluene	10	10.060	3	101
Trichloroethene	10	10.555	5	106	1,4 Dichlorobenzene	10	9.945	3	
1,2-Dichloropropane	10	9.898	2	99	n-Butylbenzene	10	10.418	3	104
Dibromomethane	10	10.430	2	104	1,2 Dichlorobenzene	10	4.755	2	95
Bromodichloromethane	10	10.473	3	105	DBCP	10	10.030	3	10
cis-1,3-Dichloropropene	10	9.520	1	95	1,2,4-Trichlorobenzene	10	8.905	1	89
Toluene	10	10.235	4	102	Hexachlorobutadiene	10	9.590	3	96
t-1,3-Dichloropropene	10	9.383	1	94	Naphthalene	10	9.985	4	100
4-Methyl-2-pentanone	50	49.858	4	100	1,2,3-Trichlorobenzene	10	9.678	2	9

16.0 DEFINITIONS

16.1 **Accuracy**: The closeness of agreement between an observed value and an accepted reference value. When applied to a set of observed values, accuracy will be a combination of a random component and of a common systematic error (or bias) component.

16.2 **Batch**: A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 20, then each group of 20 samples or less will all be handled as a separate batch.

16.3 **Bias**: The deviation due to matrix effects of the measured value (x_s - x_u) from a known spiked amount, where x_s is the spiked sample and x_u is the un-spiked sample. Bias can be assessed by comparing a measured value to an accepted reference value in a sample of known concentration or by determining the recovery of a known amount of contaminant spiked into a sample (matrix spike).

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16.4 **Control Sample**: A QC sample introduced into a process to monitor the performance of the system.

16.5 **Equipment Blank**: A sample of analyte-free media which has been used to rinse the sampling equipment. It is collected after completion of decontamination and prior to sampling. This blank is useful in documenting adequate decontamination of sampling equipment.

16.6 **Method Reporting Limit**: The lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The MRL is generally 5 to 10 times the MDL. ESS Laboratory sets the MRL to the lowest non-zero standard in the calibration curve or higher.

16.7 **Field Duplicates**: Independent samples which are collected as close as possible to the same point in space and time. They are two separate samples taken from the same source, stored in separate containers, and analyzed independently. These duplicates are useful in documenting the precision of the sampling process.

16.8 **Blank Spike (BS)**: A known matrix spiked with compound(s) representative of the target analytes. This is used to document laboratory performance.

16.9 **Matrix**: The component or substrate (e.g., surface water, drinking water) which contains the analyte of interest.

16.10 **Matrix Duplicate**: An intralaboratory split sample which is used to document the precision of a method in a given sample matrix.

16.11 **Matrix Spike**: An aliquot of sample spiked with a known concentration of target analyte(s). The spiking occurs prior to sample preparation and analysis. A matrix spike is used to document the bias of a method in a given sample matrix.

16.12 **Matrix Spike Duplicates**: Intralaboratory split samples spiked with identical concentrations of target analyte(s). The spiking occurs prior to sample preparation and analysis. They are used to document the precision and bias of a method in a given sample matrix.

16.13 **Method Blank**: An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank should be carried through the complete sample preparation and analytical procedure. The method blank is used to document contamination resulting from the analytical process.

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16.14 **Method Detection Limit (MDL)**: The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte

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concentration is greater than zero and is determined from analysis of a sample in a given matrix type containing the analyte. See SOP 110_0013 for further explanation.

16.15 **Organic-Free Reagent Water**: For volatiles, all references to water in the method refer to water in which an interferant is not observed at the method detection limit of the compounds of interest. A water purification system is used to generate organic-free deionized water.

16.16 **Records**: Include all logbooks, papers, machine readable materials, or other documentary materials, regardless of physical form or characteristics.

16.17 **Surrogate**: An organic compound which is similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which is not normally found in environmental samples.

16.18 **Trip Blank**: A sample of analyte-free media taken from the laboratory to the sampling site and returned to the laboratory unopened. A trip blank is used to document contamination attributable to shipping and field handling procedures. This type of blank is useful in documenting contamination of volatile organics samples.

17.0 PERSONNEL QUALIFICATIONS

17.1 Analysts who perform this analysis must have a working knowledge or quantitative and qualitative analysis, instrumental methods of analysis, chemical laboratory methods, and equipment.

17.2 All analysts, before performing any analysis, participate in the ESS Laboratory training program (SOP80_0016). The training process consists of reading the Standard Operating Procedure, gaining instruction on the procedure from an experienced analyst, and performing the initial demonstration of capability.

18.0 TROUBLESHOOTING

18.1 If BFB criteria are not met, the analysis must be repeated. Analysis can not begin until BFB meets acceptance criteria. Repeated failures indicate that the MS acquisition parameters must be adjusted. These parameters should be adjusted in manual tune, saved and the tune repeated.

18.2 If manual or auto-tune does not produce BFB spectra within acceptance criteria, the MS source may need cleaning. Cleaning instructions are found in the MS detector manual.

18.3 Method blanks must not contain any target compound greater than ½ the MRL with the exception of methylene chloride. If the method blank does not meet

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criteria, then system must be cleaned before processing samples. This includes washing the purge vessel and baking the trap.

18.4 See laboratory supervisor or operations manager for all other maintenance problems.

18.5 Record all maintenance in the instrument's maintenance logbook.

19.0 Data Management And Records

19.1 **Data Management** - ESS Laboratory's utilizes the Promium Element LIMS system as part of its Data Management system. Client sample information is entered into ELEMENT LIMS and analyses are assigned to each sample. The LIMS allows EPA hold times, minimum batch QC requirements, and QC criteria to be assigned to each analysis. Standards can be entered and assigned to QC samples through the LIMS. Once analysis has been performed, data is imported using DataTool avoiding manual errors. In conjunction with Crystal Reports, the ELEMENT system allows for a wide variety of reporting formats.

19.2 **Records** – The specific retention periods required in the NELAC Standards, EPA-CFR and state and local statutes are followed or exceeded. At a minimum, data records are retained for five years from last use (10 years for drinking water). If there is a question about whether a record should be retained or disposed because no specific requirement could be found, the record is retained until such time as a retention period is specified. Records are stored in specified-labeled locations and are easily retrievable. All raw data associated with testing is also retained including; computer printouts, chromatograms, review forms, and logbooks.

20.0 ATTACHMENTS

Table 2 – QC Summary for EPA Method 524.2

Attachment A – Analysis Map – VOA

Attachment B – Drinking Water Sampling Instructions using Sodium Thiosulfate

Attachment C – Drinking Water Sampling Instructions using Ascorbic Acid

Attachment D - Run Parameters

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TABLE 2
Quality Control Summary for EPA Method 524.2

QC Element	Performance standard	Corrective action
GC/MS Tune with BFB	Criteria listed in section 11.1 Performed Every 12 hours	Perform instrument maintenance, re-tune. Suspend analyses until within tune criteria.
Initial Calibration	Minimum of 5 standards Low standard at MRL. %RSD≤20% or R^2>0.99 for all compound.	Recalibrate when ICV or CCV no longer meet criteria.
Continuing Calibration	Every 12 hours prior to sample analysis. Concentration at 10 ug/L. Must contain all target analytes. %D must be ≤ 30% for all analytes, ≤50 % for poor purgers. Can use as LCS if performed using source separate from Ical	Re-analyze once with freshly prepared CCV. If still outside criteria, re-calibrate.
ICV	Performed immediately after Ical. %D must be ≤ 30% for all analytes, ≤50 % for poor purgers.	Re-analyze once with freshly prepared ICV. If still outside criteria, re-calibrate.
Method Blanks	Every 20 samples prior to sample analysis and after CCV. Matrix and preservative specific. Target analytes < ½ MRL except for common lab contaminants (such as acetone, MEK, and methylene chloride) which must be < 5x MRL.	Locate source of problem, re-analyze method blank. Re-analyze associated samples unless sample is non-detect for the contaminant. See section 11.0.
Laboratory Control Sample (LCS)	Every 12 hours prior to sample analysis. Concentration same as MS and prepared from source different than Ical. Must contain all target analytes. 70-130% Recovery	Re-analyze the LCS Locate source of problem and re-analyze associated samples.
MS/MSD	Every 20 samples Prepared from source different than Ical. Percent recoveries 70-130% RPD ≤ 20%	Check LCS If LCS recoveries acceptable, note non-conformance in narrative.
Surrogates	80-120%	If outside criteria, re-analyze sample unless obvious interference is present.
Internal standards	Area counts in sample must be between 70-130% of the areas in the associated CCV or 50-200% of the Ical. Retention times must be within ± 30s of associated CCV.	If outside criteria, re-analyze sample unless obvious interference is present.

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Attachment A

Page _____ of _____

VOA Analysis Map

Analysis _____ **ESS Work Order/Samples** _____

ESS Sample ID	Instr	Directory[1]	File #	Dil	Batch	Sequence	Cal #	RPT	Samp wt/ Vol/ Comments

Comments[2]: _____

VOC Spike ID: **Spike Amt:** **Final Volume:** _____

1 Q:/VOA/"Instr"/
2 All QC SOP compliant unless otherwise noted.
Control # 20.0027-0901a

Analyst_____ Date:____

Reviewed by:_____(second review includes a check and approval of manual integrations)

Q:\VOA\VoaWork_Map.XLS.xls

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Attachment B: Drinking Water sampling instructions using Sodium Thiosulfate

ESS Laboratory VOC 524.2 Sample Collection Procedure for samples Containing Residual Chlorine

Application: Full 524.2 Volatile analysis, this procedure does not apply to Trihalomethane samples

Number of Vials: **3 per sample**

Vial Description: VOA vial containing 3-4 drops of Sodium Thiosulfate (Na2SO3).

Materials: VOA vial containing 1:1 HCl
Plastic transfer pipette

Sampling Procedure:

If samples, such as finished drinking water or waste water, are suspected to contain residual chlorine, fill sample vial to overflowing, but take care not to flush out the Sodium thiosulfate. No air bubbles should pass through the sample as the vial is filled, or be trapped in the sample when the vial is sealed. Adjust the pH of the each sample vial to <2 by carefully adding two drops of 1:1 HCl for each 40 mL of sample. Seal the sample vials, Teflon face down, and invert three times to mix.

Sample Shipping:

Samples must be kept cool with ice or ice packs (4° +/- 2° C)

A **Trip Blank** must accompany each batch of samples. A Trip Blank is a VOA vial that has been filled in the laboratory with Type I water and contains 10% Sodium thiosulfate.

For additional information call ESS Laboratory: 401–461–7181

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Attachment C: Drinking Water sampling instructions using Ascorbic Acid

ESS Laboratory VOC 524.2 Sample Collection Procedure for samples Containing Residual Chlorine

Application: Full 524.2 Volatile analysis, this procedure does not apply to Trihalomethane samples

Number of Vials: **3 per sample**

Vial Description: VOA vial containing 25 mg of Ascorbic Acid

Materials: VOA vial containing 1:1 HCl
Plastic transfer pipette

Sampling Procedure:

If samples, such as finished drinking water or waste water, are suspected to contain residual chlorine, fill sample vial to overflowing, but take care not to flush out the ascorbic acid. No air bubbles should pass through the sample as the vial is filled, or be trapped in the sample when the vial is sealed. Adjust the pH of the each sample vial to <2 by carefully adding two drops of 1:1 HCl for each 40 mL of sample. Seal the sample vials, Teflon face down, and invert three times to mix.

Sample Shipping:

Samples must be kept cool with ice or ice packs (4° +/- 2° C)

A **Trip Blank** must accompany each batch of samples. A Trip Blank is a VOA vial that has been filled in the laboratory with Type I water and contains 25 mg of ascorbic acid.

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ATTACHMENT D

TOPLEVEL PARAMETERS

Method Information For: C:\HPCHEM\1\METHODS\DW081511.M

Method Sections To Run:

( ) Save Copy of Method With Data
( ) Pre-Run Cmd/Macro =
(X) Data Acquisition
(X) Data Analysis
( ) Post-Run Cmd/Macro =

Method Comments:
Method 524.2

END OF TOPLEVEL PARAMETERS

INSTRUMENT CONTROL PARAMETERS

Sample Inlet: GC
Injection Source: Manual
Injection Location: Front
Mass Spectrometer: Enabled

==========

HP6890 GC METHOD

==========

OVEN
Initial temp: 45 'C (On)
Initial time: 3.00 min
Maximum temp: 240 'C
Equilibration time: 0.10 min
Ramps:

#	Rate	Final temp	Final time
1	36.00	190	0.00
2	20.00	225	2.00
3	0.0(Off)		

Post temp: 0 'C
Post time: 0.00 min
Run time: 10.78 min

FRONT INLET (UNKNOWN)
Mode: Split
Initial temp: 150 'C (On)
Pressure: 12.12 psi (On)
Split ratio: 60:1
Split flow: 34.5 mL/min
Total flow: 37.1 mL/min
Gas saver: On
Saver flow: 150.0 mL/min

BACK INLET ()

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Saver time: 1.00 min
Gas type: Helium

COLUMN 1
Capillary Column
Model Number: J&W DB-VRX
Max temperature: 260 'C
Nominal length: 20.0 m
Nominal diameter: 180.00 um
Nominal film thickness: 1.00 um
Mode: constant pressure
Pressure: 12.12 psi
Nominal initial flow: 0.6 mL/min
Average velocity: 34 cm/sec
Inlet: Front Inlet
Outlet: MSD
Outlet pressure: vacuum

COLUMN 2
(not installed)

FRONT DETECTOR (NO DET)

BACK DETECTOR (NO DET)

SIGNAL 1
Data rate: 20 Hz
Type: test plot
Save Data: Off
Zero: 0.0 (Off)
Range: 0
Fast Peaks: Off
Attenuation: 0

SIGNAL 2
Data rate: 20 Hz
Type: test plot
Save Data: Off
Zero: 0.0 (Off)
Range: 0
Fast Peaks: Off
Attenuation: 0

COLUMN COMP 1
(No Detectors Installed)

COLUMN COMP 2
(No Detectors Installed)

THERMAL AUX 2
Use: MSD Transfer Line Heater
Description:
Initial temp: 250 'C (On)
Initial time: 0.00 min

#	Rate	Final temp	Final time
1	0.0	(Off)	

POST RUN
Post Time: 0.00 min

TIME TABLE

Time	Specifier	Parameter & Setpoint

7673 Injector

Front Injector:

Sample Washes	0	
Sample Pumps	0	
Injection Volume	1.0	microliters
Syringe Size	10.0	microliters
Nanoliter Adapter	Off	
PostInj Solvent A Washes	0	
PostInj Solvent B Washes	0	
Viscosity Delay	0	seconds
Plunger Speed	Fast	

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Back Injector:

Sample Washes	0	
Sample Pumps	0	
Injection Volume	1.0	microliters
Syringe Size	10.0	microliters
Nanoliter Adapter	Off	
PostInj Solvent A Washes	0	
PostInj Solvent B Washes	0	
Viscosity Delay	0	seconds
Plunger Speed	Fast	

MS ACQUISITION PARAMETERS

General Information

Tune File	: BFB1.U
Acquistion Mode	: Scan

MS Information

Solvent Delay	: 1.00 min
EM Absolute	: True
Resulting EM Voltage	: 1505.9

[Scan Parameters]

Low Mass	: 35		
High Mass	: 260		
Threshold	: 500		
Sample #	: 3	A/D Samples	8
Plot 2 low mass	: 50		
Plot 2 high mass	: 550		

[MSZones]

MS Quad	: 150 C	maximum 200 C
MS Source	: 230 C	maximum 250 C

END OF MS ACQUISITION PARAMETERS

END OF INSTRUMENT CONTROL PARAMETERS

DATA ANALYSIS PARAMETERS

Method Name: C:\HPCHEM\1\METHODS\DW081511.M

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Percent Report Settings

Sort By: Signal

Output Destination
    Screen: No
    Printer: Yes
    File: No

Integration Events: Meth Default

Generate Report During Run Method: No

Signal Correlation Window: 0.020

Qualitative Report Settings

Peak Location of Unknown: Apex

Library to Search	Minimum Quality
c:\database\nbs75k.l	0

Integration Events: Meth Default

Report Type: Summary

Output Destination
    Screen: No
    Printer: Yes
    File: No

Generate Report During Run Method: No

Quantitative Report Settings

Report Type: Summary

Output Destination
    Screen: Yes
    Printer: No
    File: No

Generate Report During Run Method: Yes

ELEMENT ID: 1108009
Calibration Last Updated: Sat Sep 03 14:22:51 2011

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Reference Window: 10.00 Percent
Non-Reference Window: 5.00 Percent
Correlation Window: 0.02 minutes
Default Multiplier: 1.00
Default Sample Concentration: 0.00

Compound Information

---

1) Fluorobenzene (ISTD)

Ret. Time 4.79 min., Extract & Integrate from 4.29 to 5.29 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 96.00			*** METH DEFAULT ***
Q1 70.00	25.40	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		5.000	1690570
.5		5.000	1682534
2		5.000	1718448
4		5.000	1747504
10		5.000	1825568
20		5.000	1912901
40		5.000	1914540
80		5.000	2066169

Qualifier Peak Analysis ON ISTD conc: 5.000 ug/l
Curve Fit: Avg. RF

---

2) Dichlorodifluoromethane ( )

Ret. Time 1.33 min., Extract & Integrate from 0.83 to 1.83 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 85.00			*** METH DEFAULT ***
Q1 87.00	32.70	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	36960
2		2.000	149394
4		4.000	293456
10		10.000	668055
20		20.000	1409318
40		40.000	2846921
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

3) Chloromethane ( )

Ret. Time 1.42 min., Extract & Integrate from 0.92 to 1.92 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 50.00			*** METH DEFAULT ***

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Q1 52.00 31.90 30.0 *** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	45391
2		2.000	166918
4		4.000	323945
10		10.000	747394
20		20.000	1603576
40		40.000	3076897
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

4) Vinyl Chloride ( )

Ret. Time 1.52 min., Extract & Integrate from 1.02 to 2.02 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 62.00			*** METH DEFAULT ***
Q1 64.00	32.50	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		0.100	11891
.5		0.500	42344
2		2.000	157435
4		4.000	282213
10		10.000	650908
20		20.000	1398475
40		40.000	-1
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Linear

---

5) Bromomethane ( )

Ret. Time 1.71 min., Extract & Integrate from 1.21 to 2.21 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 94.00			*** METH DEFAULT ***
Q1 96.00	95.00	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	34956
2		2.000	122284
4		4.000	222754
10		10.000	474121
20		20.000	961781
40		40.000	1891085
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Linear

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6) Chloroethane ( )

Ret. Time 1.79 min., Extract & Integrate from 1.29 to 2.29 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	64.00			*** METH DEFAULT ***
Q1	66.00	32.80	30.0	*** METH DEFAULT ***
Q2	49.00	25.70	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	26265
2		2.000	100426
4		4.000	196041
10		10.000	451403
20		20.000	973394
40		40.000	1888351
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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7) Trichlorofluoromethane ( )

Ret. Time 2.12 min., Extract & Integrate from 1.62 to 2.62 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	101.00			*** METH DEFAULT ***
Q1	103.00	64.70	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	54664
2		2.000	215043
4		4.000	427510
10		10.000	983929
20		20.000	2090455
40		40.000	4135057
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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8) Diethyl Ether ( )

Ret. Time 2.28 min., Extract & Integrate from 1.78 to 2.78 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	74.00			*** METH DEFAULT ***
Q1	59.00	130.40	30.0	*** METH DEFAULT ***
Q2	45.00	96.70	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	11727
2		2.000	46602
4		4.000	90300
10		10.000	210772

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20	20.000	438586
40	40.000	908276
80	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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9) Acetone ( )

Ret. Time 2.21 min., Extract & Integrate from 1.71 to 2.71 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 58.00			*** METH DEFAULT ***
Q1 43.00	350.00	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		2.500	5722
2		10.000	18760
4		20.000	35941
10		50.000	76997
20		100.000	160218
40		200.000	316000
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Linear

---

10) Methyl Iodide ( )

Ret. Time 2.48 min., Extract & Integrate from 1.98 to 2.98 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 142.00			*** METH DEFAULT ***
Q1 127.00	41.20	30.0	*** METH DEFAULT ***
Q2 141.00	14.10	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	26301
2		2.000	140389
4		4.000	329007
10		10.000	892249
20		20.000	2054017
40		40.000	4200866
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

11) 1,1-Dichloroethene ( )

Ret. Time 2.46 min., Extract & Integrate from 1.96 to 2.96 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 96.00			*** METH DEFAULT ***
Q1 61.00	164.40	30.0	*** METH DEFAULT ***

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Q2 63.00 52.80 30.0 *** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	29614
2		2.000	114139
4		4.000	220331
10		10.000	516731
20		20.000	1114541
40		40.000	2223528
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

12) Carbon Disulfide ( )

Ret. Time 2.71 min., Extract & Integrate from 2.21 to 3.21 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	76.00			*** METH DEFAULT ***
Q1	78.00	9.50	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	83841
2		2.000	321837
4		4.000	656523
10		10.000	1582942
20		20.000	3478192
40		40.000	7071409
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

13) 1,1,2-Trichloro-1,2,2-trifluoroethane ( )

Ret. Time 2.62 min., Extract & Integrate from 2.12 to 3.12 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	101.00			*** METH DEFAULT ***
Q1	151.00	88.40	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	28596
2		2.000	112978
4		4.000	228356
10		10.000	524434
20		20.000	1099626
40		40.000	2231704
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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14) Allyl Chloride ( )

Ret. Time 2.65 min., Extract & Integrate from 2.15 to 3.15 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	41.00			*** METH DEFAULT ***
Q1	39.00	75.00	30.0	*** METH DEFAULT ***
Q2	76.00	33.50	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	52801
2		2.000	207549
4		4.000	408008
10		10.000	968400
20		20.000	2103511
40		40.000	4222910
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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15) Methylene Chloride ( )

Ret. Time 2.58 min., Extract & Integrate from 2.08 to 3.08 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	84.00			*** METH DEFAULT ***
Q1	86.00	64.40	30.0	*** METH DEFAULT ***
Q2	49.00	134.70	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	36304
2		2.000	120018
4		4.000	227085
10		10.000	511490
20		20.000	1060423
40		40.000	2141972
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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16) Acrylonitrile ( )

Ret. Time 2.52 min., Extract & Integrate from 2.02 to 3.02 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	53.00			*** METH DEFAULT ***
Q1	52.00	87.80	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	3782
2		2.000	15516
4		4.000	29250
10		10.000	65748

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20	20.000	139866
40	40.000	290625
80	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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17) Methyl tert-Butyl Ether ( )

Ret. Time 3.24 min., Extract & Integrate from 2.74 to 3.74 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 73.00			*** METH DEFAULT ***
Q1 57.00	24.00	30.0	*** METH DEFAULT ***
Q2 41.00	29.50	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	40869
2		2.000	179899
4		4.000	360740
10		10.000	833379
20		20.000	1749135
40		40.000	3639731
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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18) trans-1,2-Dichloroethene ( )

Ret. Time 3.12 min., Extract & Integrate from 2.62 to 3.62 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 96.00			*** METH DEFAULT ***
Q1 61.00	145.90	30.0	*** METH DEFAULT ***
Q2 98.00	63.70	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	13826
2		2.000	125636
4		4.000	248179
10		10.000	576571
20		20.000	1246875
40		40.000	2503492
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Linear

---

19) 1,1-Dichloroethane ( )

Ret. Time 3.32 min., Extract & Integrate from 2.82 to 3.82 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 63.00			*** METH DEFAULT ***

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Q1	65.00	32.20	30.0	*** METH DEFAULT ***
Q2	83.00	12.60	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	58632
2		2.000	231190
4		4.000	456871
10		10.000	1067804
20		20.000	2258809
40		40.000	4525649
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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20) Vinyl Acetate ( )

Ret. Time 3.47 min., Extract & Integrate from 2.97 to 3.97 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	43.00			*** METH DEFAULT ***
Q1	86.00	10.30	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	28743
2		2.000	103993
4		4.000	217175
10		10.000	515102
20		20.000	1054155
40		40.000	2335063
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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21) Di-isopropyl ether ( )

Ret. Time 3.71 min., Extract & Integrate from 3.21 to 4.21 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	45.00			*** METH DEFAULT ***
Q1	43.00	55.20	30.0	*** METH DEFAULT ***
Q2	87.00	26.20	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	116487
2		2.000	369008
4		4.000	725811
10		10.000	1665410
20		20.000	3479414
40		40.000	7052270
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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22) cis-1,2 Dichloroethene ( )

Ret. Time 3.75 min., Extract & Integrate from 3.25 to 4.25 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	96.00			*** METH DEFAULT ***
Q1	61.00	128.80	30.0	*** METH DEFAULT ***
Q2	98.00	63.70	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	39534
2		2.000	132799
4		4.000	264232
10		10.000	616511
20		20.000	1302211
40		40.000	2644750
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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23) 2,2-Dichloropropane ( )

Ret. Time 3.95 min., Extract & Integrate from 3.45 to 4.45 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	77.00			*** METH DEFAULT ***
Q1	97.00	19.40	30.0	*** METH DEFAULT ***
Q2	41.00	74.60	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	45851
2		2.000	169003
4		4.000	332646
10		10.000	796515
20		20.000	1744258
40		40.000	3518798
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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24) 2-Butanone ( )

Ret. Time 3.69 min., Extract & Integrate from 3.19 to 4.19 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	72.00			*** METH DEFAULT ***
Q1	43.00	464.70	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		2.500	7666
2		10.000	25258

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4	20.000	48472
10	50.000	109303
20	100.000	242685
40	200.000	498826
80	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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25) Methyl Acrylate ( )

Ret. Time 3.98 min., Extract & Integrate from 3.48 to 4.48 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 55.00			*** METH DEFAULT ***
Q1 85.00	15.40	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	13314
2	2.000	50262
4	4.000	99671
10	10.000	228442
20	20.000	482427
40	40.000	998052
80	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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26) Methylacrylonitrile ( )

Ret. Time 3.74 min., Extract & Integrate from 3.24 to 4.24 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 41.00			*** METH DEFAULT ***
Q1 67.00	59.10	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	10951
2	2.000	31644
4	4.000	58470
10	10.000	130631
20	20.000	293087
40	40.000	621971
80	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Linear

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27) Bromochloromethane ( )

Ret. Time 3.86 min., Extract & Integrate from 3.36 to 4.36 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 128.00			*** METH DEFAULT ***

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Q1	49.00	159.80	30.0	*** METH DEFAULT ***
Q2	130.00	130.80	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	14550
2		2.000	47300
4		4.000	97133
10		10.000	220117
20		20.000	460966
40		40.000	954488
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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28) Ethyl tertiary-butyl ether ( )

Ret. Time 3.99 min., Extract & Integrate from 3.49 to 4.49 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	59.00			*** METH DEFAULT ***
Q1	41.00	27.00	30.0	*** METH DEFAULT ***
Q2	87.00	43.00	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	79145
2		2.000	266041
4		4.000	537069
10		10.000	1231033
20		20.000	2566449
40		40.000	5330292
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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29) Tetrahydrofuran ( )

Ret. Time 4.14 min., Extract & Integrate from 3.64 to 4.64 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	42.00			*** METH DEFAULT ***
Q1	72.00	37.90	30.0	*** METH DEFAULT ***
Q2	41.00	58.50	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	4732
2		2.000	15733
4		4.000	31939
10		10.000	67195
20		20.000	132552
40		40.000	321731
80		not used for this compound	

Qualifier Peak Analysis ON

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Curve Fit: Avg. RF

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30) Chloroform ( )

Ret. Time 3.90 min., Extract & Integrate from 3.40 to 4.40 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	83.00			*** METH DEFAULT ***
Q1	85.00	65.00	30.0	*** METH DEFAULT ***
Q2	47.00	26.00	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	67293
2		2.000	219876
4		4.000	434950
10		10.000	1009787
20		20.000	2143181
40		40.000	4311061
80		80.000	8994486

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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31) 1,1,1-Trichloroethane ( )

Ret. Time 4.40 min., Extract & Integrate from 3.90 to 4.90 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	97.00			*** METH DEFAULT ***
Q1	99.00	64.00	30.0	*** METH DEFAULT ***
Q2	61.00	50.00	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	50331
2		2.000	190189
4		4.000	431996
10		10.000	1053058
20		20.000	2349823
40		40.000	4797589
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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32) 1,1-Dichloropropene ( )

Ret. Time 4.52 min., Extract & Integrate from 4.02 to 5.02 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	75.00			*** METH DEFAULT ***
Q1	110.00	34.70	30.0	*** METH DEFAULT ***
Q2	77.00	30.90	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	

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.5	0.500	52176	
2	2.000	215991	
4	4.000	429289	
10	10.000	1027420	
20	20.000	2246127	
40	40.000	4561147	
80	not used for this compound		

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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33) 1-Chlorobutane ( )

Ret. Time 4.56 min., Extract & Integrate from 4.06 to 5.06 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 56.00			*** METH DEFAULT ***
Q1 41.00	62.90	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	70255
2	2.000	292591
4	4.000	586952
10	10.000	1387207
20	20.000	3029018
40	40.000	6105790
80	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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34) Carbon Tetrachloride ( )

Ret. Time 4.62 min., Extract & Integrate from 4.12 to 5.12 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 117.00			*** METH DEFAULT ***
Q1 119.00	95.20	30.0	*** METH DEFAULT ***
Q2 121.00	30.70	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	42586
2	2.000	176418
4	4.000	372946
10	10.000	931815
20	20.000	2144871
40	40.000	4499321
80	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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35) Benzene ( )

Ret. Time 4.66 min., Extract & Integrate from 4.16 to 5.16 min.

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Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	78.00			*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	160427
2		2.000	645737
4		4.000	1294586
10		10.000	3021310
20		20.000	6440595
40		40.000	12339306
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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36) 1,2-Dichloroethane ( )

Ret. Time 4.34 min., Extract & Integrate from 3.84 to 4.84 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	62.00			*** METH DEFAULT ***
Q1	98.00	9.60	30.0	*** METH DEFAULT ***
Q2	49.00	29.40	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	36374
2		2.000	118014
4		4.000	278606
10		10.000	598304
20		20.000	1333259
40		40.000	2706295
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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37) Tertiary-amyl methyl ether ( )

Ret. Time 4.79 min., Extract & Integrate from 4.29 to 5.29 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	73.00			*** METH DEFAULT ***
Q1	43.00	37.00	30.0	*** METH DEFAULT ***
Q2	87.00	23.30	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	77542
2		2.000	245638
4		4.000	474123
10		10.000	1076359
20		20.000	2299783
40		40.000	4864454
80		not used for this compound	

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Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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38) Trichloroethene ( )

Ret. Time 5.05 min., Extract & Integrate from 4.55 to 5.55 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 95.00			*** METH DEFAULT ***
Q1 97.00	63.40	30.0	*** METH DEFAULT ***
Q2 130.00	110.40	30.0	*** METH DEFAULT ***
Q3 132.00	108.20	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	42036
2		2.000	165582
4		4.000	323560
10		10.000	764549
20		20.000	1678117
40		40.000	3404760
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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39) 1,2-Dichloropropane ( )

Ret. Time 5.02 min., Extract & Integrate from 4.52 to 5.52 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 63.00			*** METH DEFAULT ***
Q1 112.00	5.00	30.0	*** METH DEFAULT ***
Q2 61.00	14.60	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	34953
2		2.000	139627
4		4.000	283987
10		10.000	668898
20		20.000	1426581
40		40.000	2985914
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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40) Dibromomethane ( )

Ret. Time 5.00 min., Extract & Integrate from 4.50 to 5.50 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 93.00			*** METH DEFAULT ***
Q1 95.00	84.00	30.0	*** METH DEFAULT ***
Q2 174.00	114.20	30.0	*** METH DEFAULT ***

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Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	12269
2		2.000	48235
4		4.000	102153
10		10.000	238889
20		20.000	515181
40		40.000	1077910
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

41) Methyl Methacrylate ( )

Ret. Time 5.21 min., Extract & Integrate from 4.71 to 5.71 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	69.00			*** METH DEFAULT ***
Q1	41.00	152.10	30.0	*** METH DEFAULT ***
Q2	100.00	38.80	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	10014
2		2.000	39731
4		4.000	85246
10		10.000	200209
20		20.000	439437
40		40.000	954233
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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42) 1,4-Dioxane ( )

Ret. Time 5.18 min., Extract & Integrate from 4.68 to 5.68 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	88.00			*** METH DEFAULT ***
Q1	58.00	66.10	30.0	*** METH DEFAULT ***
Q2	43.00	32.90	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		10.000	3291
2		40.000	13800
4		80.000	26106
10		200.000	64098
20		400.000	135144
40		800.000	284023
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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43) Bromodichloromethane ( )

Ret. Time 5.08 min., Extract & Integrate from 4.58 to 5.58 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 83.00			*** METH DEFAULT ***
Q1 85.00	66.20	30.0	*** METH DEFAULT ***
Q2 127.00	9.20	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	33933
2		2.000	137452
4		4.000	302081
10		10.000	741238
20		20.000	1655844
40		40.000	3542073
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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44) 2-Nitropropane ( )

Ret. Time 5.07 min., Extract & Integrate from 4.57 to 5.57 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 43.00			*** METH DEFAULT ***
Q1 41.00	91.50	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	2900
2		2.000	10592
4		4.000	23547
10		10.000	54007
20		20.000	116495
40		40.000	277934
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

45) cis-1,3-Dichloropropene ( )

Ret. Time 5.46 min., Extract & Integrate from 4.96 to 5.96 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 75.00			*** METH DEFAULT ***
Q1 77.00	32.30	30.0	*** METH DEFAULT ***
Q2 39.00	59.20	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		0.100	8289
.5		0.500	31105
2		2.000	139660
4		4.000	305908
10		10.000	775254

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20	20.000	1779959
40	40.000	3871476
80	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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46) Toluene ( )

Ret. Time 5.88 min., Extract & Integrate from 5.38 to 6.38 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 92.00			*** METH DEFAULT ***
Q1 91.00	170.70	30.0	*** METH DEFAULT ***
Q2 65.00	20.10	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	96196
2	2.000	391179
4	4.000	800115
10	10.000	1921826
20	20.000	4182210
40	40.000	8381625
80	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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47) trans-1,3-Dichloropropene ( )

Ret. Time 5.70 min., Extract & Integrate from 5.20 to 6.20 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 75.00			*** METH DEFAULT ***
Q1 77.00	31.60	30.0	*** METH DEFAULT ***
Q2 39.00	55.80	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	0.100	5943
.5	0.500	21572
2	2.000	94464
4	4.000	213249
10	10.000	559050
20	20.000	1305048
40	40.000	2892875
80	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

48) 4-Methyl-2-pentanone ( )

Ret. Time 5.54 min., Extract & Integrate from 5.04 to 6.04 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 58.00			*** METH DEFAULT ***

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Q1	43.00	275.10	30.0	*** METH DEFAULT ***
Q2	85.00	43.30	30.0	*** METH DEFAULT ***
Q3	100.00	39.20	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		2.500	24697
2		10.000	103203
4		20.000	210621
10		50.000	482279
20		100.000	1043113
40		200.000	2251644
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

49) Ethyl Methacrylate ( )

Ret. Time 5.95 min., Extract & Integrate from 5.45 to 6.45 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	69.00			*** METH DEFAULT ***
Q1	41.00	81.70	30.0	*** METH DEFAULT ***
Q2	99.00	17.60	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	19903
2		2.000	83504
4		4.000	172830
10		10.000	417422
20		20.000	928791
40		40.000	2041841
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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50) 1,1,2-Trichloroethane ( )

Ret. Time 5.78 min., Extract & Integrate from 5.28 to 6.28 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	83.00			*** METH DEFAULT ***
Q1	97.00	118.00	30.0	*** METH DEFAULT ***
Q2	85.00	63.90	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	16002
2		2.000	60484
4		4.000	123861
10		10.000	284482
20		20.000	609828
40		40.000	1279606
80		not used for this compound	

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Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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51) 1,3-Dichloropropane ( )

Ret. Time 5.91 min., Extract & Integrate from 5.41 to 6.41 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 76.00			*** METH DEFAULT ***
Q1 78.00	32.00	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	37545
2		2.000	144945
4		4.000	295111
10		10.000	684312
20		20.000	1459201
40		40.000	3043375
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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52) Tetrachloroethene ( )

Ret. Time 6.26 min., Extract & Integrate from 5.76 to 6.76 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 164.00			*** METH DEFAULT ***
Q1 129.00	83.70	30.0	*** METH DEFAULT ***
Q2 131.00	81.70	30.0	*** METH DEFAULT ***
Q3 166.00	128.90	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	43581
2		2.000	179022
4		4.000	345147
10		10.000	790720
20		20.000	1936176
40		40.000	3506601
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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53) 2-Hexanone ( )

Ret. Time 6.01 min., Extract & Integrate from 5.51 to 6.51 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 43.00			*** METH DEFAULT ***
Q1 58.00	48.70	30.0	*** METH DEFAULT ***
Q2 57.00	18.20	30.0	*** METH DEFAULT ***
Q3 100.00	11.00	30.0	*** METH DEFAULT ***

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Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		2.500	49467
2		10.000	200526
4		20.000	392586
10		50.000	899015
20		100.000	1979397
40		200.000	4157200
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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54) Dibromochloromethane ( )

Ret. Time 6.04 min., Extract & Integrate from 5.54 to 6.54 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	129.00			*** METH DEFAULT ***
Q1	127.00	77.30	30.0	*** METH DEFAULT ***
Q2	131.00	24.60	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		0.100	4709
.5		0.500	17627
2		2.000	71941
4		4.000	158920
10		10.000	402267
20		20.000	924702
40		40.000	2036515
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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55) 1,2-Dibromoethane ( )

Ret. Time 6.17 min., Extract & Integrate from 5.67 to 6.67 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	107.00			*** METH DEFAULT ***
Q1	109.00	94.60	30.0	*** METH DEFAULT ***
Q2	188.00	4.30	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	15080
2		2.000	61744
4		4.000	130132
10		10.000	305515
20		20.000	673239
40		40.000	1438591
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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56) Chlorobenzene ( )

Ret. Time 6.61 min., Extract & Integrate from 6.11 to 7.11 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 112.00			*** METH DEFAULT ***
Q1 77.00	59.60	30.0	*** METH DEFAULT ***
Q2 114.00	32.40	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	109324
2		2.000	432293
4		4.000	868996
10		10.000	2026834
20		20.000	4298947
40		40.000	8681321
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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57) 1,1,1,2-Tetrachloroethane ( )

Ret. Time 6.57 min., Extract & Integrate from 6.07 to 7.07 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 131.00			*** METH DEFAULT ***
Q1 133.00	95.80	30.0	*** METH DEFAULT ***
Q2 119.00	62.00	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	27157
2		2.000	114376
4		4.000	242600
10		10.000	600647
20		20.000	1357287
40		40.000	2922262
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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58) Ethylbenzene ( )

Ret. Time 6.71 min., Extract & Integrate from 6.21 to 7.21 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 91.00			*** METH DEFAULT ***
Q1 106.00	32.00	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	188023
2		2.000	788584
4		4.000	1612705
10		10.000	3765172

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20	20.000	7977305
40	40.000	13344160
80	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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59) Xylene P,M ( )

Ret. Time 6.81 min., Extract & Integrate from 6.31 to 7.31 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 106.00			*** METH DEFAULT ***
Q1 91.00	192.90	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	0.200	39207
.5	1.000	149852
2	4.000	629108
4	8.000	1266040
10	20.000	2966623
20	40.000	6318075
40	80.000	12261430
80	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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60) Xylene O ( )

Ret. Time 7.01 min., Extract & Integrate from 6.51 to 7.51 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 106.00			*** METH DEFAULT ***
Q1 91.00	211.00	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	0.100	20530
.5	0.500	74477
2	2.000	312937
4	4.000	627585
10	10.000	1472227
20	20.000	3145819
40	40.000	6466312
80	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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61) Styrene ( )

Ret. Time 6.97 min., Extract & Integrate from 6.47 to 7.47 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 104.00			*** METH DEFAULT ***
Q1 78.00	43.90	30.0	*** METH DEFAULT ***

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Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	112400
2		2.000	442769
4		4.000	897779
10		10.000	2123551
20		20.000	4603885
40		40.000	9365958
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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62) Bromoform ( )

Ret. Time 6.86 min., Extract & Integrate from 6.36 to 7.36 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	173.00			*** METH DEFAULT ***
Q1	175.00	50.30	30.0	*** METH DEFAULT ***
Q2	254.00	14.40	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		0.100	2449
.5		0.500	9798
2		2.000	37330
4		4.000	80546
10		10.000	197613
20		20.000	472889
40		40.000	1103963
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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63) Isopropylbenzene ( )

Ret. Time 7.18 min., Extract & Integrate from 6.68 to 7.68 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	105.00			*** METH DEFAULT ***
Q1	120.00	28.10	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	202767
2		2.000	836722
4		4.000	1682632
10		10.000	3935012
20		20.000	8273088
40		40.000	13561272
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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64) Bromofluorobenzene (SURR) ( )

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Ret. Time 7.20 min., Extract & Integrate from 6.70 to 7.70 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 95.00			*** METH DEFAULT ***
Q1 174.00	89.00	30.0	*** METH DEFAULT ***
Q2 176.00	86.40	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	5.000	527028
.5	5.000	553882
2	5.000	589917
4	5.000	605718
10	5.000	628334
20	5.000	653945
40	5.000	683757
80	5.000	727953

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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65) 1,2,3-Trichloropropane ( )

Ret. Time 7.07 min., Extract & Integrate from 6.57 to 7.57 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 75.00			*** METH DEFAULT ***
Q1 77.00	32.10	30.0	*** METH DEFAULT ***
Q2 110.00	36.00	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	17780
2	2.000	70126
4	4.000	142224
10	10.000	324133
20	20.000	732606
40	40.000	1606352
80	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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66) Bromobenzene ( )

Ret. Time 7.31 min., Extract & Integrate from 6.81 to 7.81 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 156.00			*** METH DEFAULT ***
Q1 77.00	145.40	30.0	*** METH DEFAULT ***
Q2 158.00	97.00	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	45940
2	2.000	171950
4	4.000	346822
10	10.000	794531

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20	20.000	1696454
40	40.000	3609305
80	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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67) 1,1,2,2-Tetrachloroethane ( )

Ret. Time 7.00 min., Extract & Integrate from 6.50 to 7.50 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 83.00			*** METH DEFAULT ***
Q1 131.00	10.80	30.0	*** METH DEFAULT ***
Q2 85.00	65.70	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	0.100	6625
.5	0.500	19972
2	2.000	75157
4	4.000	142720
10	10.000	322705
20	20.000	687452
40	40.000	1501234
80	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Linear

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68) trans-1,4-dichloro-2-Butene ( )

Ret. Time 7.08 min., Extract & Integrate from 6.58 to 7.58 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 89.00			*** METH DEFAULT ***
Q1 77.00	83.60	30.0	*** METH DEFAULT ***
Q2 75.00	247.20	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	1403
2	2.000	7354
4	4.000	15162
10	10.000	40075
20	20.000	106495
40	40.000	-1
80	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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69) n-Propylbenzene ( )

Ret. Time 7.40 min., Extract & Integrate from 6.90 to 7.90 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 91.00			*** METH DEFAULT ***

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Q1 120.00 24.40 30.0 *** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	232625
2		2.000	959571
4		4.000	1957894
10		10.000	4503677
20		20.000	9315222
40		40.000	14828613
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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70) 2-Chlorotoluene ( )

Ret. Time 7.45 min., Extract & Integrate from 6.95 to 7.95 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 91.00			*** METH DEFAULT ***
Q1 126.00	35.00	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	152808
2		2.000	580413
4		4.000	1104284
10		10.000	2647671
20		20.000	5646676
40		40.000	11189058
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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71) 4-Chlorotoluene ( )

Ret. Time 7.49 min., Extract & Integrate from 6.99 to 7.99 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 91.00			*** METH DEFAULT ***
Q1 126.00	34.40	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	145869
2		2.000	555187
4		4.000	1140849
10		10.000	2490011
20		20.000	5157242
40		40.000	10504085
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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72) 1,3,5-Trimethylbenzene ( )

Ret. Time 7.54 min., Extract & Integrate from 7.04 to 8.04 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	105.00			*** METH DEFAULT ***
Q1	120.00	51.50	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	165875
2		2.000	692952
4		4.000	1399212
10		10.000	3272803
20		20.000	6889088
40		40.000	12771475
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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73) tert-Butylbenzene ( )

Ret. Time 7.69 min., Extract & Integrate from 7.19 to 8.19 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	119.00			*** METH DEFAULT ***
Q1	91.00	63.90	30.0	*** METH DEFAULT ***
Q2	134.00	27.00	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	159871
2		2.000	650834
4		4.000	1321966
10		10.000	3080440
20		20.000	6581018
40		40.000	12539394
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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74) Pentachloroethane ( )

Ret. Time 7.59 min., Extract & Integrate from 7.09 to 8.09 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	165.00			*** METH DEFAULT ***
Q1	167.00	129.30	30.0	*** METH DEFAULT ***
Q2	117.00	109.80	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	12343
2		2.000	46411
4		4.000	102656
10		10.000	267484

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20	20.000	470943
40	40.000	-1
80	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

75) 1,2,4-Trimethylbenzene ( )

Ret. Time 7.74 min., Extract & Integrate from 7.24 to 8.24 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 105.00			*** METH DEFAULT ***
Q1 120.00	48.30	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	171825
2		2.000	691642
4		4.000	1395733
10		10.000	3242121
20		20.000	6862349
40		40.000	13151847
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

76) sec-Butylbenzene ( )

Ret. Time 7.80 min., Extract & Integrate from 7.30 to 8.30 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 105.00			*** METH DEFAULT ***
Q1 134.00	22.50	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	225678
2		2.000	937207
4		4.000	1889398
10		10.000	4329423
20		20.000	9047247
40		40.000	14688099
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

77) 1,3 Dichlorobenzene ( )

Ret. Time 7.84 min., Extract & Integrate from 7.34 to 8.34 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 146.00			*** METH DEFAULT ***
Q1 111.00	37.80	30.0	*** METH DEFAULT ***
Q2 148.00	63.40	30.0	*** METH DEFAULT ***

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Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	98691
2		2.000	401233
4		4.000	755591
10		10.000	1675027
20		20.000	3612788
40		40.000	7418009
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

78) 4-Isopropyltoluene ( )

Ret. Time 7.89 min., Extract & Integrate from 7.39 to 8.39 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 119.00			*** METH DEFAULT ***
Q1 134.00	27.10	30.0	*** METH DEFAULT ***
Q2 91.00	23.10	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	185017
2		2.000	773128
4		4.000	1582978
10		10.000	3680162
20		20.000	7753964
40		40.000	13898481
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

79) 1,4 Dichlorobenzene ( )

Ret. Time 7.88 min., Extract & Integrate from 7.38 to 8.38 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 146.00			*** METH DEFAULT ***
Q1 111.00	35.70	30.0	*** METH DEFAULT ***
Q2 148.00	65.40	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	102707
2		2.000	340485
4		4.000	690244
10		10.000	1593261
20		20.000	3342611
40		40.000	7094610
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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80) Hexachloroethane ( )

Ret. Time 8.34 min., Extract & Integrate from 7.84 to 8.84 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 201.00			*** METH DEFAULT ***
Q1 166.00	87.20	30.0	*** METH DEFAULT ***
Q2 117.00	108.00	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	19465
2		2.000	87418
4		4.000	187323
10		10.000	499068
20		20.000	1222429
40		40.000	2795522
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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81) n-Butylbenzene ( )

Ret. Time 8.10 min., Extract & Integrate from 7.60 to 8.60 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 91.00			*** METH DEFAULT ***
Q1 92.00	55.30	30.0	*** METH DEFAULT ***
Q2 134.00	30.40	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	154801
2		2.000	643897
4		4.000	1315328
10		10.000	3050251
20		20.000	6409277
40		40.000	12761514
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

82) 1,2-Dichlorobenzene-d4 ( )

Ret. Time 8.05 min., Extract & Integrate from 7.55 to 8.55 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 152.15			*** METH DEFAULT ***
Q1 150.15	166.50	30.0	*** METH DEFAULT ***
Q2 115.15	58.60	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		5.000	508403
.5		5.000	544533
2		5.000	565874

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4	5.000	590994
10	5.000	602868
20	5.000	627811
40	5.000	663563
80	5.000	720117

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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83) 1,2 Dichlorobenzene ( )

Ret. Time 8.06 min., Extract & Integrate from 7.56 to 8.56 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 146.00			*** METH DEFAULT ***
Q1 111.00	39.60	30.0	*** METH DEFAULT ***
Q2 148.00	65.50	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	91420
2	2.000	326167
4	4.000	629183
10	10.000	1398812
20	20.000	2972663
40	40.000	6221630
80	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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84) 1,2-Dibromo-3-Chloropropane ( )

Ret. Time 8.31 min., Extract & Integrate from 7.81 to 8.81 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 75.00			*** METH DEFAULT ***
Q1 155.00	87.10	30.0	*** METH DEFAULT ***
Q2 157.00	111.20	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	2547
2	2.000	9762
4	4.000	20032
10	10.000	46884
20	20.000	111912
40	40.000	259327
80	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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85) 1,3,5-Trichlorobenzene ( )

Ret. Time 8.76 min., Extract & Integrate from 8.26 to 9.26 min.

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Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	180.00			*** METH DEFAULT ***
Q1	182.00	97.20	30.0	*** METH DEFAULT ***
Q2	145.00	27.10	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	70443
2		2.000	248682
4		4.000	490427
10		10.000	1136746
20		20.000	2465208
40		40.000	5296527
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

86) 1,2,4-Trichlorobenzene ( )

Ret. Time 9.07 min., Extract & Integrate from 8.57 to 9.57 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	180.00			*** METH DEFAULT ***
Q1	182.00	95.10	30.0	*** METH DEFAULT ***
Q2	145.00	27.80	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	62590
2		2.000	223696
4		4.000	436329
10		10.000	998621
20		20.000	2161944
40		40.000	4690096
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

87) Hexachlorobutadiene ( )

Ret. Time 9.25 min., Extract & Integrate from 8.75 to 9.75 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	225.00			*** METH DEFAULT ***
Q1	223.00	62.70	30.0	*** METH DEFAULT ***
Q2	227.00	65.00	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		0.100	12825
.5		0.500	35249
2		2.000	147567
4		4.000	297982
10		10.000	683397
20		20.000	1476173
40		40.000	3273159
80		not used for this compound	

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Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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88) Naphthalene ( )

Ret. Time 9.22 min., Extract & Integrate from 8.72 to 9.72 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 128.00			*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	81853
2		2.000	292288
4		4.000	585992
10		10.000	1349661
20		20.000	2942248
40		40.000	6471198
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

89) 1,2,3-Trichlorobenzene ( )

Ret. Time 9.35 min., Extract & Integrate from 8.85 to 9.85 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 180.00			*** METH DEFAULT ***
Q1 182.00	95.70	30.0	*** METH DEFAULT ***
Q2 145.00	29.40	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	53349
2		2.000	189403
4		4.000	372802
10		10.000	848609
20		20.000	1831399
40		40.000	4003204
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

END OF DATA ANALYSIS PARAMETERS

Thu Sep 08 15:01:03 2011

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COLUMN 1
Capillary Column
Model Number: J&W DB-VRX
Max temperature: 260 'C
Nominal length: 20.0 m
Nominal diameter: 180.00 um
Nominal film thickness: 1.00 um
Mode: constant pressure
Pressure: 28.64 psi
Nominal initial flow: 1.5 mL/min
Average velocity: 55 cm/sec
Inlet: Front Inlet
Outlet: MSD
Outlet pressure: vacuum

COLUMN 2
(not installed)

FRONT DETECTOR (NO DET)

BACK DETECTOR (NO DET)

SIGNAL 1
Data rate: 20 Hz
Type: test plot
Save Data: Off
Zero: 0.0 (Off)
Range: 0
Fast Peaks: Off
Attenuation: 0

SIGNAL 2
Data rate: 20 Hz
Type: test plot
Save Data: Off
Zero: 0.0 (Off)
Range: 0
Fast Peaks: Off
Attenuation: 0

COLUMN COMP 1
(No Detectors Installed)

COLUMN COMP 2
(No Detectors Installed)

THERMAL AUX 2
Use: MSD Transfer Line Heater
Description:
Initial temp: 280 'C (On)
Initial time: 0.00 min

#	Rate	Final temp	Final time
1		0.0(Off)	

POST RUN
Post Time: 0.00 min

TIME TABLE

Time	Specifier	Parameter & Setpoint

7673 Injector

Front Injector:

Sample Washes	0	
Sample Pumps	0	
Injection Volume	1.0	microliters
Syringe Size	10.0	microliters
Nanoliter Adapter	Off	
PostInj Solvent A Washes	0	
PostInj Solvent B Washes	0	
Viscosity Delay	0	seconds
Plunger Speed	Fast	

Back Injector:

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Sample Washes	0
Sample Pumps	0
Injection Volume	1.0 microliters
Syringe Size	10.0 microliters
Nanoliter Adapter	Off
PostInj Solvent A Washes	0
PostInj Solvent B Washes	0
Viscosity Delay	0 seconds
Plunger Speed	Fast

MS ACQUISITION PARAMETERS

General Information

Tune File : BFB.U
Acquistion Mode : Scan

MS Information

Solvent Delay : 0.50 min

EM Absolute : True
Resulting EM Voltage : 1352.9

[Scan Parameters]

Low Mass : 35
High Mass : 260
Threshold : 500
Sample # : 4 A/D Samples 16
Plot 2 low mass : 50
Plot 2 high mass : 550

[MSZones]

MS Quad : 150 C maximum 200 C
MS Source : 230 C maximum 250 C

END OF MS ACQUISITION PARAMETERS

END OF INSTRUMENT CONTROL PARAMETERS

DATA ANALYSIS PARAMETERS

Method Name: C:\HPCHEM\1\METHODS\DW121107.M

MASTER

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Percent Report Settings

Sort By: Signal

Output Destination
    Screen: No
    Printer: Yes
    File: No

Integration Events: Meth Default

Generate Report During Run Method: No

Signal Correlation Window: 0.020

Qualitative Report Settings

Peak Location of Unknown: Apex

Library to Search	Minimum Quality
c:\database\nbs75k.l	0

Integration Events: Meth Default

Report Type: Summary

Output Destination
    Screen: No
    Printer: Yes
    File: No

Generate Report During Run Method: No

Quantitative Report Settings

Report Type: Summary

Output Destination
    Screen: Yes
    Printer: Yes
    File: No

Generate Report During Run Method: Yes

ELEMENT ID: 0710008
Calibration Last Updated: Fri Dec 14 13:11:04 2007

Reference Window: 10.00 Percent
Non-Reference Window: 5.00 Percent

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Correlation Window: 0.02 minutes
Default Multiplier: 1.00
Default Sample Concentration: 0.00

Compound Information

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1) Fluorobenzene (ISTD)

Ret. Time 3.72 min., Extract & Integrate from 3.22 to 4.22 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 96.00			*** METH DEFAULT ***
Q1 77.00	22.10	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	5.000	3033916
.5	5.000	2817685
2	5.000	2868259
4	5.000	2906919
10	5.000	2955227
20	5.000	3315924
40	5.000	3457593
80	5.000	3371630

Qualifier Peak Analysis ON ISTD conc: 5.000 ug/l
Curve Fit: Avg. RF

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2) Dichlorodifluoromethane ( )

Ret. Time 0.79 min., Extract & Integrate from 0.29 to 1.29 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 85.00			*** METH DEFAULT ***
Q1 87.00	32.50	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	31332
2	2.000	122286
4	4.000	270669
10	10.000	650037
20	20.000	1316798
40	40.000	2705084
80	80.000	4849089

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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3) Chloromethane ( )

Ret. Time 0.84 min., Extract & Integrate from 0.34 to 1.34 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 50.00			*** METH DEFAULT ***
Q1 52.00	32.60	30.0	*** METH DEFAULT ***

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Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	31157
2		2.000	109487
4		4.000	295145
10		10.000	710391
20		20.000	1353030
40		40.000	3420633
80		80.000	5504260

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

4) Vinyl Chloride ( )

Ret. Time 0.89 min., Extract & Integrate from 0.39 to 1.39 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 62.00			*** METH DEFAULT ***
Q1 64.00	30.50	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		0.100	7067
.5		0.500	25561
2		2.000	105700
4		4.000	214052
10		10.000	533840
20		20.000	1132807
40		40.000	1900927
80		80.000	-1

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

5) Bromomethane ( )

Ret. Time 1.01 min., Extract & Integrate from 0.51 to 1.51 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 94.00			*** METH DEFAULT ***
Q1 96.00	90.70	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	14883
2		2.000	61310
4		4.000	119238
10		10.000	284703
20		20.000	598393
40		40.000	1198724
80		80.000	1977293

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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6) Chloroethane ( )

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Ret. Time 1.06 min., Extract & Integrate from 0.56 to 1.56 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 64.00			*** METH DEFAULT ***
Q1 66.00	32.80	30.0	*** METH DEFAULT ***
Q2 49.00	31.00	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	14250
2	2.000	57081
4	4.000	123175
10	10.000	287706
20	20.000	598018
40	40.000	1197318
80	80.000	2043237

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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7) Trichlorofluoromethane ( )

Ret. Time 1.25 min., Extract & Integrate from 0.75 to 1.75 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 101.00			*** METH DEFAULT ***
Q1 103.00	64.50	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	34584
2	2.000	143084
4	4.000	313090
10	10.000	789160
20	20.000	1737098
40	40.000	3526725
80	80.000	6142972

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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8) Diethyl Ether ( )

Ret. Time 1.35 min., Extract & Integrate from 0.85 to 1.85 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 74.00			*** METH DEFAULT ***
Q1 59.00	163.10	30.0	*** METH DEFAULT ***
Q2 45.00	139.40	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	5089
2	2.000	19226
4	4.000	41043
10	10.000	99068
20	20.000	244788
40	40.000	536924

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80	80.000	1003652

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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9) Acetone ( )

Ret. Time 1.30 min., Extract & Integrate from 0.80 to 1.80 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 58.00			*** METH DEFAULT ***
Q1 43.00	455.70	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	2.500	2449
2	10.000	9682
4	20.000	17614
10	50.000	45045
20	100.000	104247
40	200.000	237982
80	400.000	496854

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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10) Methyl Iodide ( )

Ret. Time 1.48 min., Extract & Integrate from 0.98 to 1.98 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 142.00			*** METH DEFAULT ***
Q1 127.00	58.20	30.0	*** METH DEFAULT ***
Q2 141.00	14.50	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	16873
2	2.000	69111
4	4.000	144238
10	10.000	359852
20	20.000	733260
40	40.000	1523178
80	80.000	2495939

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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11) 1,1-Dichloroethene ( )

Ret. Time 1.46 min., Extract & Integrate from 0.96 to 1.96 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 96.00			*** METH DEFAULT ***
Q1 61.00	266.60	30.0	*** METH DEFAULT ***
Q2 63.00	83.40	30.0	*** METH DEFAULT ***

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Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	12013
2		2.000	50142
4		4.000	114346
10		10.000	279654
20		20.000	685285
40		40.000	1516319
80		80.000	2805853

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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12) Carbon Disulfide ( )

Ret. Time 1.61 min., Extract & Integrate from 1.11 to 2.11 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	76.00			*** METH DEFAULT ***
Q1	78.00	9.10	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	54773
2		2.000	227728
4		4.000	476891
10		10.000	1186209
20		20.000	2693364
40		40.000	5537404
80		80.000	9558042

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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13) 1,1,2-Trichloro-1,2,2-trifluoroethane ( )

Ret. Time 1.56 min., Extract & Integrate from 1.06 to 2.06 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	101.00			*** METH DEFAULT ***
Q1	151.00	69.00	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	12021
2		2.000	46688
4		4.000	99120
10		10.000	248258
20		20.000	648997
40		40.000	1560204
80		80.000	2929839

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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14) Allyl Chloride ( )

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Ret. Time 1.57 min., Extract & Integrate from 1.07 to 2.07 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 41.00			*** METH DEFAULT ***
Q1 39.00	81.30	30.0	*** METH DEFAULT ***
Q2 76.00	25.70	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	34976
2	2.000	139375
4	4.000	298824
10	10.000	734930
20	20.000	1782451
40	40.000	3884984
80	80.000	6891420

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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15) Methylene Chloride ( )

Ret. Time 1.53 min., Extract & Integrate from 1.03 to 2.03 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 84.00			*** METH DEFAULT ***
Q1 86.00	63.40	30.0	*** METH DEFAULT ***
Q2 49.00	174.20	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	21281
2	2.000	69958
4	4.000	132791
10	10.000	313715
20	20.000	664694
40	40.000	1386441
80	80.000	2605325

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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16) Acrylonitrile ( )

Ret. Time 1.49 min., Extract & Integrate from 0.99 to 1.99 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 53.00			*** METH DEFAULT ***
Q1 52.00	80.60	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	1587
2	2.000	8482
4	4.000	17260
10	10.000	43371
20	20.000	99687
40	40.000	225584

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80	80.000	466418

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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17) Methyl tert-Butyl Ether ( )

Ret. Time 1.93 min., Extract & Integrate from 1.43 to 2.43 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 73.00			*** METH DEFAULT ***
Q1 [illegible]	23.00	30.0	*** METH DEFAULT ***
Q2 [illegible]	31.00	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	27474
2	2.000	102811
4	4.000	209175
10	10.000	491788
20	20.000	1125091
40	40.000	2622416
80	80.000	5452244

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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18) trans-1,2-Dichloroethene ( )

Ret. Time 1.85 min., Extract & Integrate from 1.35 to 2.35 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt [illegible]			*** METH DEFAULT ***
Q1 [illegible]	223.30	30.0	*** METH DEFAULT ***
Q2 [illegible]	62.90	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	12826
2	2.000	50194
4	4.000	110111
10	10.000	261910
20	20.000	628136
40	40.000	1397512
80	80.000	2833769

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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19) 1,1-Dichloroethane ( )

Ret. Time 1.99 min., Extract & Integrate from 1.49 to 2.49 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 63.00			*** METH DEFAULT ***
Q1 65.00	31.50	30.0	*** METH DEFAULT ***
Q2 83.00	11.80	30.0	*** METH DEFAULT ***

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Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	34660
2		2.000	132259
4		4.000	279701
10		10.000	678205
20		20.000	1579869
40		40.000	3456163
80		80.000	6817187

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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20) Vinyl Acetate ( )

Ret. Time 2.12 min., Extract & Integrate from 1.62 to 2.62 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 43.00			*** METH DEFAULT ***
Q1 86.00	5.60	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	18181
2		2.000	72167
4		4.000	140440
10		10.000	349855
20		20.000	816515
40		40.000	1965356
80		80.000	4043601

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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21) Di-isopropyl ether ( )

Ret. Time 2.34 min., Extract & Integrate from 1.84 to 2.84 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 45.00			*** METH DEFAULT ***
Q1 43.00	54.20	30.0	*** METH DEFAULT ***
Q2 87.00	18.00	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	63818
2		2.000	253754
4		4.000	528629
10		10.000	1288781
20		20.000	3031529
40		40.000	6406725
80		80.000	11482441

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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22) cis-1,2 Dichloroethene ( )

Ret. Time 2.38 min., Extract & Integrate from 1.88 to 2.88 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 96.00			*** METH DEFAULT ***
Q1 61.00	193.30	30.0	*** METH DEFAULT ***
Q2 98.00	62.70	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	14078
2	2.000	53634
4	4.000	111049
10	10.000	263945
20	20.000	613927
40	40.000	1353429
80	80.000	2800492

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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23) 2,2-Dichloropropane ( )

Ret. Time 2.60 min., Extract & Integrate from 2.10 to 3.10 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 77.00			*** METH DEFAULT ***
Q1 97.00	17.90	30.0	*** METH DEFAULT ***
Q2 41.00	63.40	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	25869
2	2.000	100110
4	4.000	235066
10	10.000	591975
20	20.000	1623971
40	40.000	4247692
80	80.000	8409848

Qualifier Peak Analysis ON
Curve Fit: Linear

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24) 2-Butanone ( )

Ret. Time 2.31 min., Extract & Integrate from 1.81 to 2.81 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 72.00			*** METH DEFAULT ***
Q1 43.00	689.70	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	2.500	1547
2	10.000	10460
4	20.000	19835
10	50.000	54110

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20	100.000	131310
40	200.000	331502
80	400.000	750914

Qualifier Peak Analysis ON
Curve Fit: Linear

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25) Methyl Acrylate ( )

Ret. Time 3.41 min., Extract & Integrate from 2.91 to 3.91 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 85.00			*** METH DEFAULT ***
Q1 85.00	14.70	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	15232
2	2.000	65074
4	4.000	145982
10	10.000	350644
20	20.000	864079
40	40.000	2117269
80	80.000	4346878

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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26) Methylacrylonitrile ( )

Ret. Time 2.36 min., Extract & Integrate from 1.86 to 2.86 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 41.00			*** METH DEFAULT ***
Q1 67.00	18.50	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	3554
2	2.000	13452
4	4.000	35595
10	10.000	75007
20	20.000	157223
40	40.000	-1
80	80.000	-1

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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27) Bromochloromethane ( )

Ret. Time 2.50 min., Extract & Integrate from 2.00 to 3.00 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 128.00			*** METH DEFAULT ***
Q1 49.00	191.60	30.0	*** METH DEFAULT ***
Q2 130.00	129.60	30.0	*** METH DEFAULT ***

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Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	6886
2		2.000	19141
4		4.000	52553
10		10.000	147040
20		20.000	359263
40		40.000	687283
80		80.000	-1

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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28) Ethyl tertiary-butyl ether ( )

Ret. Time 2.65 min., Extract & Integrate from 2.15 to 3.15 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 59.00			*** METH DEFAULT ***
Q1 41.00	21.60	30.0	*** METH DEFAULT ***
Q2 87.00	38.20	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	56677
2		2.000	196812
4		4.000	422791
10		10.000	1037258
20		20.000	2466713
40		40.000	5194375
80		80.000	9578866

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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29) Tetrahydrofuran ( )

Ret. Time 2.83 min., Extract & Integrate from 2.33 to 3.33 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 42.00			*** METH DEFAULT ***
Q1 72.00	39.10	30.0	*** METH DEFAULT ***
Q2 41.00	55.70	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	2668
2		2.000	8373
4		4.000	15331
10		10.000	39005
20		20.000	89966
40		40.000	210650
80		80.000	472056

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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30) Chloroform ( )

Ret. Time 2.54 min., Extract & Integrate from 2.04 to 3.04 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 83.00			*** METH DEFAULT ***
Q1 85.00	63.10	30.0	*** METH DEFAULT ***
Q2 47.00	26.70	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	38728
2	2.000	119790
4	4.000	312154
10	10.000	774069
20	20.000	1840830
40	40.000	3923193
80	80.000	6446963

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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31) 1,1,1-Trichloroethane ( )

Ret. Time 4.81 min., Extract & Integrate from 4.31 to 5.31 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 97.00			*** METH DEFAULT ***
Q1 99.00	63.40	30.0	*** METH DEFAULT ***
Q2 61.00	64.20	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	14140
2	2.000	57782
4	4.000	120388
10	10.000	300464
20	20.000	688973
40	40.000	1516201
80	80.000	3170508

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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32) 1,1-Dichloropropene ( )

Ret. Time 4.46 min., Extract & Integrate from 3.96 to 4.96 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 75.00			*** METH DEFAULT ***
Q1 110.00	19.30	30.0	*** METH DEFAULT ***
Q2 77.00	31.50	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	29134
2	2.000	122316

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4	4.000	268159
10	10.000	691239
20	20.000	1659594
40	40.000	3681174
80	80.000	7390794

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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33) 1-Chlorobutane ( )

Ret. Time 3.41 min., Extract & Integrate from 2.91 to 3.91 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt [illegible]			*** METH DEFAULT ***
Q1 [illegible]	59.10	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	40829
2	2.000	175515
4	4.000	391117
10	10.000	955283
20	20.000	2336398
40	40.000	5695758
80	80.000	11403424

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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34) Carbon Tetrachloride ( )

Ret. Time 3.48 min., Extract & Integrate from 2.98 to 3.98 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt [illegible]			*** METH DEFAULT ***
Q1 [illegible]	94.70	30.0	*** METH DEFAULT ***
Q2 [illegible]	30.70	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	24687
2	2.000	106566
4	4.000	233912
10	10.000	586654
20	20.000	1553519
40	40.000	-1
80	80.000	-1

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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35) Benzene ( )

Ret. Time 3.53 min., Extract & Integrate from 3.03 to 4.03 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration

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Tgt 78.00 *** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	108632
2		2.000	430521
4		4.000	923313
10		10.000	2269155
20		20.000	5239596
40		40.000	10625262
80		80.000	16209715

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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36) 1,1-Dichloroethane ( )

Ret. Time 3.12 min., Extract & Integrate from 2.62 to 3.62 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 62.00			*** METH DEFAULT ***
Q1 98.00	7.10	30.0	*** METH DEFAULT ***
Q2 49.00	28.00	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	22310
2		2.000	88144
4		4.000	180900
10		10.000	445816
20		20.000	1065986
40		40.000	2313058
80		80.000	4590567

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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37) Tertiary-amyl methyl ether ( )

Ret. Time 3.71 min., Extract & Integrate from 3.21 to 4.21 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 73.00			*** METH DEFAULT ***
Q1 43.00	33.10	30.0	*** METH DEFAULT ***
Q2 87.00	23.00	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	78915
2		2.000	200961
4		4.000	376443
10		10.000	875306
20		20.000	2038681
40		40.000	4419668
80		80.000	8592300

Qualifier Peak Analysis ON
Curve Fit: Linear

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38) Trichloroethene ( )

Ret. Time 4.00 min., Extract & Integrate from 3.50 to 4.50 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 95.00			*** METH DEFAULT ***
Q1 97.00	64.30	30.0	*** METH DEFAULT ***
Q2 130.00	83.40	30.0	*** METH DEFAULT ***
Q3 132.00	79.30	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	29105
2	2.000	109494
4	4.000	237956
10	10.000	604797
20	20.000	1413279
40	40.000	3073250
80	80.000	5821030

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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39) 1,2-Dichloropropane ( )

Ret. Time 3.97 min., Extract & Integrate from 3.47 to 4.47 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 63.00			*** METH DEFAULT ***
Q1 112.00	3.80	30.0	*** METH DEFAULT ***
Q2 61.00	13.80	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	28217
2	2.000	104601
4	4.000	215517
10	10.000	512026
20	20.000	1166358
40	40.000	2553892
80	80.000	4985269

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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40) Dibromomethane ( )

Ret. Time 3.93 min., Extract & Integrate from 3.43 to 4.43 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 93.00			*** METH DEFAULT ***
Q1 95.00	84.70	30.0	*** METH DEFAULT ***
Q2 174.00	78.50	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	

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.5	0.500	8937
2	2.000	36450
4	4.000	74293
10	10.000	182877
20	20.000	437511
40	40.000	952752
80	80.000	1951527

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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41) Methyl Methacrylate ( )

Ret. Time 4.22 min., Extract & Integrate from 3.72 to 4.72 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt [illegible]			*** METH DEFAULT ***
Q1 [illegible]	129.40	30.0	*** METH DEFAULT ***
Q2 [illegible]	36.30	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	6424
2		2.000	28467
4		4.000	62844
10		10.000	167303
20		20.000	410847
40		40.000	994684
80		80.000	2053373

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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42) 1,4-Dioxane ( )

Ret. Time 4.14 min., Extract & Integrate from 3.64 to 4.64 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt [illegible]			*** METH DEFAULT ***
Q1 [illegible]	84.50	30.0	*** METH DEFAULT ***
Q2 [illegible]	36.10	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		10.000	324
2		40.000	4698
4		80.000	10710
10		200.000	28726
20		400.000	68133
40		800.000	151064
80		1600.000	297019

Qualifier Peak Analysis ON
Curve Fit: Linear

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43) Bromodichloromethane ( )

Ret. Time 4.03 min., Extract & Integrate from 3.53 to 4.53 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 83.00			*** METH DEFAULT ***
Q1 85.00	64.50	30.0	*** METH DEFAULT ***
Q2 127.00	8.10	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	24898
2	2.000	101803
4	4.000	223259
10	10.000	536192
20	20.000	1313914
40	40.000	3177622
80	80.000	6598428

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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44) 2-Nitropropane ( )

Ret. Time 4.04 min., Extract & Integrate from 3.54 to 4.54 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 43.00			*** METH DEFAULT ***
Q1 41.00	90.40	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	857
2	2.000	3688
4	4.000	7711
10	10.000	22460
20	20.000	65526
40	40.000	195469
80	80.000	526451

Qualifier Peak Analysis ON
Curve Fit: Quadratic

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45) cis-1,3-Dichloropropene ( )

Ret. Time 4.46 min., Extract & Integrate from 3.96 to 4.96 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 75.00			*** METH DEFAULT ***
Q1 77.00	31.50	30.0	*** METH DEFAULT ***
Q2 39.00	55.80	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	0.100	7425
.5	0.500	29134
2	2.000	122316
4	4.000	268159
10	10.000	691239
20	20.000	1659594
40	40.000	3681174

80	80.000	7390794

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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46) Toluene ( )

Ret. Time 4.92 min., Extract & Integrate from 4.42 to 5.42 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 92.00			*** METH DEFAULT ***
Q1 91.00	168.60	30.0	*** METH DEFAULT ***
Q2 65.00	18.00	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	83022
2		2.000	354336
4		4.000	756770
10		10.000	1943415
20		20.000	4210818
40		40.000	8504001
80		80.000	11884977

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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47) trans-1,3-Dichloropropene ( )

Ret. Time 4.74 min., Extract & Integrate from 4.24 to 5.24 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 75.00			*** METH DEFAULT ***
Q1 77.00	31.80	30.0	*** METH DEFAULT ***
Q2 39.00	47.40	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		0.100	5090
.5		0.500	19741
2		2.000	92096
4		4.000	211830
10		10.000	564509
20		20.000	1432926
40		40.000	3248349
80		80.000	6473680

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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48) 4-Methyl-2-pentanone ( )

Ret. Time 4.57 min., Extract & Integrate from 4.07 to 5.07 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 58.00			*** METH DEFAULT ***
Q1 43.00	284.60	30.0	*** METH DEFAULT ***
Q2 85.00	34.20	30.0	*** METH DEFAULT ***

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Q3 100.00 26.60 30.0 *** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		2.500	14865
2		10.000	70616
4		20.000	151811
10		50.000	411846
20		100.000	1005852
40		200.000	2442832
80		400.000	4889324

Qualifier Peak Analysis ON
Curve Fit: Linear

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49) Ethyl Methacrylate ( )

Ret. Time 5.02 min., Extract & Integrate from 4.52 to 5.52 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 69.00			*** METH DEFAULT ***
Q1 41.00	92.70	30.0	*** METH DEFAULT ***
Q2 99.00	13.20	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	-1
2		2.000	70276
4		4.000	154911
10		10.000	433140
20		20.000	1044298
40		40.000	2410765
80		80.000	4865111

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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50) 1,1,2-Trichloroethane ( )

Ret. Time 4.81 min., Extract & Integrate from 4.31 to 5.31 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 83.00			*** METH DEFAULT ***
Q1 97.00	115.50	30.0	*** METH DEFAULT ***
Q2 85.00	65.30	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	11757
2		2.000	51008
4		4.000	107221
10		10.000	267330
20		20.000	603003
40		40.000	1325551
80		80.000	2787104

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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51) 1,3-Dichloropropane ( )

Ret. Time 4.95 min., Extract & Integrate from 4.45 to 5.45 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 76.00			*** METH DEFAULT ***
Q1 78.00	33.00	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	29346
2	2.000	123101
4	4.000	255428
10	10.000	631020
20	20.000	1469382
40	40.000	3177709
80	80.000	6409200

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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52) Tetrachloroethene ( )

Ret. Time 5.31 min., Extract & Integrate from 4.81 to 5.81 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 164.00			*** METH DEFAULT ***
Q1 129.00	95.00	30.0	*** METH DEFAULT ***
Q2 131.00	92.20	30.0	*** METH DEFAULT ***
Q3 166.00	126.50	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	30549
2	2.000	128565
4	4.000	304078
10	10.000	780279
20	20.000	1836799
40	40.000	4283658
80	80.000	7511298

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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53) 2-Hexanone ( )

Ret. Time 5.07 min., Extract & Integrate from 4.57 to 5.57 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 43.00			*** METH DEFAULT ***
Q1 58.00	52.30	30.0	*** METH DEFAULT ***
Q2 57.00	17.90	30.0	*** METH DEFAULT ***
Q3 100.00	9.00	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	

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.5	2.500	27011
2	10.000	131644
4	20.000	289106
10	50.000	787212
20	100.000	1851402
40	200.000	4236312
80	400.000	7539620

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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54) Dibromochloromethane ( )

Ret. Time 5.07 min., Extract & Integrate from 4.57 to 5.57 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 129.00			*** METH DEFAULT ***
Q1 127.00	77.20	30.0	*** METH DEFAULT ***
Q2 131.00	24.30	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		0.100	3077
.5		0.500	12991
2		2.000	61375
4		4.000	131396
10		10.000	356406
20		20.000	846178
40		40.000	1973175
80		80.000	4217462

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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55) 1,2-Dibromoethane ( )

Ret. Time 5.19 min., Extract & Integrate from 4.69 to 5.69 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 107.00			*** METH DEFAULT ***
Q1 109.00	93.60	30.0	*** METH DEFAULT ***
Q2 188.00	2.00	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	12919
2		2.000	55661
4		4.000	117401
10		10.000	305324
20		20.000	715174
40		40.000	1556303
80		80.000	3255634

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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56) Chlorobenzene ( )

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Ret. Time 5.65 min., Extract & Integrate from 5.15 to 6.15 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 112.00			*** METH DEFAULT ***
Q1 77.00	79.10	30.0	*** METH DEFAULT ***
Q2 114.00	32.10	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	90122
2	2.000	365313
4	4.000	785507
10	10.000	1970078
20	20.000	4313073
40	40.000	8541466
80	80.000	11704384

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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57) 1,1,1,2-Tetrachloroethane ( )

Ret. Time 5.62 min., Extract & Integrate from 5.12 to 6.12 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 131.00			*** METH DEFAULT ***
Q1 133.00	94.80	30.0	*** METH DEFAULT ***
Q2 119.00	63.00	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	20673
2	2.000	90159
4	4.000	202233
10	10.000	535512
20	20.000	1280134
40	40.000	2935922
80	80.000	5725099

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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58) Ethylbenzene ( )

Ret. Time 5.77 min., Extract & Integrate from 5.27 to 6.27 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 91.00			*** METH DEFAULT ***
Q1 106.00	31.40	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	161160
2	2.000	685677
4	4.000	1508279
10	10.000	3770498
20	20.000	7873752
40	40.000	11149258

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80	80.000	-1

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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59) Xylene P,M ( )

Ret. Time 5.87 min., Extract & Integrate from 5.37 to 6.37 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 106.00			*** METH DEFAULT ***
Q1 91.00	202.80	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	1.000	114013
2	4.000	485527
4	8.000	1060558
10	20.000	2717851
20	40.000	5873938
40	80.000	11076742
80	160.000	-1

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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60) Xylene O ( )

Ret. Time 6.06 min., Extract & Integrate from 5.56 to 6.56 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 106.00			*** METH DEFAULT ***
Q1 91.00	214.00	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	54337
2	2.000	231464
4	4.000	493290
10	10.000	1264666
20	20.000	2791658
40	40.000	5774262
80	80.000	8905597

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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61) Styrene ( )

Ret. Time 6.03 min., Extract & Integrate from 5.53 to 6.53 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 104.00			*** METH DEFAULT ***
Q1 78.00	38.30	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	

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.5	0.500	73630
2	2.000	330195
4	4.000	711583
10	10.000	1891192
20	20.000	4203942
40	40.000	8324207
80	80.000	-1

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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62) Bromoform ( )

Ret. Time 5.89 min., Extract & Integrate from 5.39 to 6.39 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 173.00			*** METH DEFAULT ***
Q1 173.00	48.30	30.0	*** METH DEFAULT ***
Q2 254.00	7.70	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	5553
2	2.000	28532
4	4.000	63305
10	10.000	180189
20	20.000	441185
40	40.000	1058296
80	80.000	2064692

Qualifier Peak Analysis ON
Curve Fit: Linear

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63) Isopropylbenzene ( )

Ret. Time 6.25 min., Extract & Integrate from 5.75 to 6.75 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 105.00			*** METH DEFAULT ***
Q1 120.00	28.20	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	147350
2	2.000	638325
4	4.000	1394619
10	10.000	3535968
20	20.000	7420548
40	40.000	-1
80	80.000	-1

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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64) Bromofluorobenzene (SURR) ( )

Ret. Time 6.25 min., Extract & Integrate from 5.75 to 6.75 min.

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Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	95.00			*** METH DEFAULT ***
Q1	174.00	41.90	30.0	*** METH DEFAULT ***
Q2	176.00	40.00	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		5.000	556575
2		5.000	605642
4		5.000	585388
10		5.000	613660
20		5.000	659987
40		5.000	704906
80		5.000	705556

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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65) 1,2,3-Trichloropropane ( )

Ret. Time 6.13 min., Extract & Integrate from 5.63 to 6.63 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	75.00			*** METH DEFAULT ***
Q1	77.00	32.00	30.0	*** METH DEFAULT ***
Q2	110.00	34.30	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	12150
2		2.000	50809
4		4.000	111182
10		10.000	303078
20		20.000	619808
40		40.000	1455200
80		80.000	2604221

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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66) Bromobenzene ( )

Ret. Time 6.34 min., Extract & Integrate from 5.84 to 6.84 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	156.00			*** METH DEFAULT ***
Q1	77.00	222.40	30.0	*** METH DEFAULT ***
Q2	158.00	96.30	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	29166
2		2.000	117524
4		4.000	246605
10		10.000	630757
20		20.000	1421789
40		40.000	3025896

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80 80.000 5597158

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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67) 1,1,2,2-Tetrachloroethane ( )

Ret. Time 6.05 min., Extract & Integrate from 5.55 to 6.55 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 83.00			*** METH DEFAULT ***
Q1 131.00	6.90	30.0	*** METH DEFAULT ***
Q2 85.00	63.80	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	0.100	3844
.5	0.500	15382
2	2.000	66017
4	4.000	136992
10	10.000	358859
20	20.000	830291
40	40.000	1791803
80	80.000	3090820

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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68) trans-1,4-dichloro-2-Butene ( )

Ret. Time 5.99 min., Extract & Integrate from 5.49 to 6.49 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 89.00			*** METH DEFAULT ***
Q1 77.00	74.80	30.0	*** METH DEFAULT ***
Q2 75.00	228.30	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	638
2	2.000	4512
4	4.000	10708
10	10.000	32385
20	20.000	84356
40	40.000	226788
80	80.000	439064

Qualifier Peak Analysis ON
Curve Fit: Linear

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69) n-Propylbenzene ( )

Ret. Time 6.45 min., Extract & Integrate from 5.95 to 6.95 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 91.00			*** METH DEFAULT ***
Q1 120.00	18.80	30.0	*** METH DEFAULT ***

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Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	159408
2		2.000	752792
4		4.000	1500131
10		10.000	3970324
20		20.000	8194754
40		40.000	11233565
80		80.000	-1

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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70) 2-Chlorotoluene ( )

Ret. Time 6.49 min., Extract & Integrate from 5.99 to 6.99 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 91.00			*** METH DEFAULT ***
Q1 126.00	36.40	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	100962
2		2.000	368309
4		4.000	920999
10		10.000	2313968
20		20.000	4582811
40		40.000	8952763
80		80.000	-1

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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71) 4-Chlorotoluene ( )

Ret. Time 6.52 min., Extract & Integrate from 6.02 to 7.02 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 91.00			*** METH DEFAULT ***
Q1 126.00	24.70	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	96491
2		2.000	411583
4		4.000	864866
10		10.000	2208525
20		20.000	4938537
40		40.000	9466229
80		80.000	-1

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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72) 1,3,5-Trimethylbenzene ( )

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Ret. Time 6.60 min., Extract & Integrate from 6.10 to 7.10 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 105.00			*** METH DEFAULT ***
Q1 120.00	48.80	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	107563
2	2.000	464083
4	4.000	1035966
10	10.000	2682494
20	20.000	5779808
40	40.000	8844332
80	80.000	-1

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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73) Tert-Butylbenzene ( )

Ret. Time 6.72 min., Extract & Integrate from 6.22 to 7.22 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 119.00			*** METH DEFAULT ***
Q1 91.00	66.00	30.0	*** METH DEFAULT ***
Q2 134.00	25.80	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	92303
2	2.000	400197
4	4.000	886626
10	10.000	2344875
20	20.000	5153945
40	40.000	8603110
80	80.000	-1

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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74) Pentachloroethane ( )

Ret. Time 6.61 min., Extract & Integrate from 6.11 to 7.11 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 165.00			*** METH DEFAULT ***
Q1 167.00	129.80	30.0	*** METH DEFAULT ***
Q2 117.00	112.70	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	8301
2	2.000	34243
4	4.000	66818
10	10.000	181308
20	20.000	411621
40	40.000	790841

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80 80.000 1473353

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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75) 1,2,4-Trimethylbenzene ( )

Ret. Time 6.78 min., Extract & Integrate from 6.28 to 7.28 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 105.00			*** METH DEFAULT ***
Q1 120.00	49.50	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	102812
2	2.000	452143
4	4.000	977137
10	10.000	2540050
20	20.000	5450487
40	40.000	9864930
80	80.000	-1

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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76) sec-Butylbenzene ( )

Ret. Time 6.82 min., Extract & Integrate from 6.32 to 7.32 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 105.00			*** METH DEFAULT ***
Q1 134.00	20.60	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	144115
2	2.000	612799
4	4.000	1345547
10	10.000	3492656
20	20.000	7462943
40	40.000	10181768
80	80.000	-1

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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77) 1,3 Dichlorobenzene ( )

Ret. Time 6.84 min., Extract & Integrate from 6.34 to 7.34 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 146.00			*** METH DEFAULT ***
Q1 111.00	42.00	30.0	*** METH DEFAULT ***
Q2 148.00	63.80	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response

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.1	not used for this compound	
.5	0.500	55013
2	2.000	215324
4	4.000	466146
10	10.000	1251420
20	20.000	2580851
40	40.000	5690137
80	80.000	9083215

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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78) 4-Isopropyltoluene ( )

Ret. Time 6.92 min., Extract & Integrate from 6.42 to 7.42 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 119.00			*** METH DEFAULT ***
Q1 134.00	27.50	30.0	*** METH DEFAULT ***
Q2 91.00	22.90	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	110802
2	2.000	489433
4	4.000	1077882
10	10.000	2772073
20	20.000	5947033
40	40.000	8693769
80	80.000	-1

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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79) 1,4 Dichlorobenzene ( )

Ret. Time 6.87 min., Extract & Integrate from 6.37 to 7.37 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 146.00			*** METH DEFAULT ***
Q1 111.00	45.00	30.0	*** METH DEFAULT ***
Q2 148.00	63.10	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	52504
2	2.000	210511
4	4.000	446875
10	10.000	1119457
20	20.000	2616981
40	40.000	5337468
80	80.000	9010711

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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80) Hexachloroethane ( )

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Ret. Time 7.28 min., Extract & Integrate from 6.78 to 7.78 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 201.00			*** METH DEFAULT ***
Q1 166.00	115.50	30.0	*** METH DEFAULT ***
Q2 117.00	171.80	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	9264
2	2.000	42836
4	4.000	103881
10	10.000	312878
20	20.000	766128
40	40.000	1844662
80	80.000	3345618

Qualifier Peak Analysis ON
Curve Fit: Linear

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81) n-Butylbenzene ( )

Ret. Time 7.10 min., Extract & Integrate from 6.60 to 7.60 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 91.00			*** METH DEFAULT ***
Q1 92.00	55.10	30.0	*** METH DEFAULT ***
Q2 134.00	23.70	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	101116
2	2.000	433773
4	4.000	992649
10	10.000	2536325
20	20.000	5408499
40	40.000	8720395
80	80.000	-1

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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82) 1,2-Dichlorobenzene-d4 ( )

Ret. Time 7.03 min., Extract & Integrate from 6.53 to 7.53 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 152.15			*** METH DEFAULT ***
Q1 150.15	163.20	30.0	*** METH DEFAULT ***
Q2 115.15	66.50	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	5.000	348724
2	5.000	377760
4	5.000	361903
10	5.000	386518

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20	5.000	403921
40	5.000	422550
80	5.000	436092

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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83) 1,2 Dichlorobenzene ( )

Ret. Time 7.04 min., Extract & Integrate from 6.54 to 7.54 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 146.00			*** METH DEFAULT ***
Q1 111.00	45.20	30.0	*** METH DEFAULT ***
Q2 148.00	64.10	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	43588
2	2.000	175301
4	4.000	368649
10	10.000	949377
20	20.000	2057969
40	40.000	4447623
80	80.000	7504978

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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84) 1,2-Dibromo-3-Chloropropane ( )

Ret. Time 7.25 min., Extract & Integrate from 6.75 to 7.75 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 75.00			*** METH DEFAULT ***
Q1 155.00	49.80	30.0	*** METH DEFAULT ***
Q2 157.00	62.20	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	1126
2	2.000	6299
4	4.000	13345
10	10.000	38267
20	20.000	82780
40	40.000	185095
80	80.000	376237

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

85) 1,2,4-Trichlorobenzene ( )

Ret. Time 7.93 min., Extract & Integrate from 7.43 to 8.43 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 180.00			*** METH DEFAULT ***

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Q1	182.00	95.30	30.0	*** METH DEFAULT ***
Q2	145.00	28.00	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	22255
2		2.000	91716
4		4.000	196945
10		10.000	524338
20		20.000	1161093
40		40.000	2630837
80		80.000	5813290

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

86) Hexachlorobutadiene ( )

Ret. Time 8.09 min., Extract & Integrate from 7.59 to 8.59 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 225.00			*** METH DEFAULT ***
Q1 223.00	63.70	30.0	*** METH DEFAULT ***
Q2 227.00	63.20	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		0.100	4451
.5		0.500	14881
2		2.000	64952
4		4.000	144414
10		10.000	374262
20		20.000	816664
40		40.000	1894579
80		80.000	4344336

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

87) Naphthalene ( )

Ret. Time 8.05 min., Extract & Integrate from 7.55 to 8.55 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 128.00			*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	28047
2		2.000	126064
4		4.000	282471
10		10.000	780602
20		20.000	1749514
40		40.000	3917418
80		80.000	8089070

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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88) 1,2,3-Trichlorobenzene ( )

Ret. Time 8.15 min., Extract & Integrate from 7.65 to 8.65 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 180.00			*** METH DEFAULT ***
Q1 182.00	95.50	30.0	*** METH DEFAULT ***
Q2 145.00	37.20	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	18230
2	2.000	76131
4	4.000	161446
10	10.000	420837
20	20.000	946961
40	40.000	2195208
80	80.000	4897589

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

END OF DATA ANALYSIS PARAMETERS

Mon Dec 17 14:51:23 2007

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Designation: D 287 – 92 (Reapproved 2006)

An American National Standard

Standard Test Method for API Gravity of Crude Petroleum and Petroleum Products (Hydrometer Method)[1]

This standard is issued under the fixed designation D 287; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense. This test method has been adopted for use by government agencies to replace Method 401 of Federal Test Method Standard No. 791b.

1. Scope

1.1 This test method covers the determination by means of a glass hydrometer of the API gravity of crude petroleum and petroleum products normally handled as liquids and having a Reid vapor pressure (Test Method D 323) of 26 psi (180 kPa) or less. Gravities are determined at 60°F (15.56°C), or converted to values at 60°F, by means of standard tables. These tables are not applicable to nonhydrocarbons or essentially pure hydrocarbons such as the aromatics.

NOTE 1—The international version of this test method is described in Test Method D 1298.

1.2 The values stated in inch-pound units are to be regarded as standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific hazard statements, see 8.3.

2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 323 Test Method for Vapor Pressure of Petroleum Products (Reid Method)

D 1250 Guide for Use of the Petroleum Measurement Tables

D 1298 Test Method for Density, Relative Density (Specific Gravity), or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method

E 1 Specification for ASTM Liquid-in-Glass Thermometers

E 100 Specification for ASTM Hydrometers

2.2 *IP Standards:*[3]

Specifications for IP Standard Thermometers

IP Specifications for Petroleum Hydrometers

3. Terminology

3.1 *Definitions:*

3.1.1 *API gravity*—a special function of relative density (specific gravity) 60/60°F (15.56/15.56°C), represented by:

$$\text{API gravity, deg} = (141.5/\text{sp gr } 60/60°\text{F}) - 131.5 \quad (1)$$

No statement of reference temperature is required, since 60°F is included in the definition.

4. Summary of Test Method

4.1 This test method is based on the principle that the gravity of a liquid varies directly with the depth of immersion of a body floating in it. The floating body, which is graduated by API gravity units in this method, is called an API hydrometer.

4.2 The API gravity is read by observing the freely floating API hydrometer and noting the graduation nearest to the apparent intersection of the horizontal plane surface of the liquid with the vertical scale of the hydrometer, after temperature equilibrium has been reached. The temperature of the sample is read from a separate accurate ASTM thermometer in the sample or from the thermometer which is an integral part of the hydrometer (thermohydrometer).

5. Significance and Use

5.1 Accurate determination of the gravity of petroleum and its products is necessary for the conversion of measured volumes to volumes at the standard temperature of 60°F (15.56°C).

5.2 Gravity is a factor governing the quality of crude oils. However, the gravity of a petroleum product is an uncertain indication of its quality. Correlated with other properties,

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.02.0A on Temperature, Density, Physical Properties.

Current edition approved May 1, 2006. Published June 2006. Originally approved in 1928. Last previous edition approved in 2000 as D 287 – 92 (2000)ε1.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from the Institute of Petroleum, 61 New Cavendish St., London WIM, 8AR, England.

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gravity can be used to give approximate hydrocarbon composition and heat of combustion.

6. Apparatus

6.1 *Hydrometers*, of glass, graduated in degrees API as listed in Table 1 and conforming to Specification E 100, or the IP Specifications for Petroleum Hydrometers.

6.2 *Thermometers*, having a range from −5 to +215°F and conforming to the requirements for Thermometer 12F as prescribed in Specification E 1 or Thermometer 64F of the Specifications for IP Standard Thermometers. A thermometer is not required if a thermohydrometer is employed.

NOTE 2—The ASTM Gravity Thermometer 12F has 0.5°F subdivisions and allowable ±0.25°F scale error. The thermometers incorporated in thermohydrometers have 2°F subdivisions and allowable ±1°F scale error.

6.3 *Hydrometer Cylinders*, of metal, clear glass, or plastic. For convenience in pouring, the cylinder may have a lip on the rim. The inside diameter of the cylinder shall be at least 25 mm greater than the outside diameter of the hydrometer used in it. The height of the cylinder shall be such that the length of the column of sample it contains is greater by at least 25 mm than the portion of the hydrometer which is immersed beneath the surface of the sample. For field testing, a sampling thief of suitable dimensions may be used.

7. Temperature of Test

7.1 The gravity determined by the hydrometer method is most accurate at or near the standard temperature of 60°F (15.56°C). Use this or any other temperature between 0 and 195°F (−18 and + 90°C) for the test, so far as it is consistent with the type of sample and necessary limiting conditions shown in Table 2.

8. Procedure

8.1 For referee testing, use the long plain form of hydrometer (1H to 10H). For field testing, use the thermohydrometer.

8.2 Adjust the temperature of the sample in accordance with Table 2. For field testing, test temperatures other than those listed in Table 2 may be used. The hydrometer cylinder shall be approximately the same temperature as the sample to be tested.

8.3 Transfer the sample into the clean hydrometer cylinder without splashing, so as to avoid the formation of air bubbles and to reduce to a minimum the evaporation of the lower boiling constituents of the more volatile samples. (**Warning—** Extremely flammable. Vapors may cause flash fire.) For the more volatile samples, transfer to the hydrometer cylinder by siphoning. (**Warning—**Do not start the siphon by mouth.) Use a rubber aspirator bulb. Remove any air bubbles formed, after they have collected on the surface of the sample, by touching them with a piece of clean filter paper before inserting the hydrometer. For field testing, make the gravity measurement directly in the sampling thief. Place the cylinder containing the sample in a vertical position in a location free from air currents. Take precautions to prevent the temperature of the sample from changing appreciably during the time necessary to complete the test. During this period, the temperature of the surrounding medium should not change more than 5°F (2°C).

8.4 Lower the hydrometer gently into the sample and, when it has settled, depress it about two scale divisions into the liquid and then release it; keep the rest of the stem dry, as unnecessary liquid on the stem changes the effective weight of the instrument, and so affects the reading obtained. With samples of low viscosity, a slight spin imparted to the instrument on releasing assists in bringing it to rest, floating freely away from the walls of the hydrometer cylinder. Allow sufficient time for the hydrometer to become completely stationary and for all air bubbles to come to the surface. This is particularly necessary in the case of the more viscous samples.

8.5 When the hydrometer has come to rest, floating freely, and the temperature of the sample is constant to 0.2°F (0.1°C), read the hydrometer to the nearest scale division. The correct reading is that point on the hydrometer scale at which the surface of the liquid cuts the scale. Determine this point by placing the eye slightly below the level of the liquid and slowly raising it until the surface, first seen as a distorted ellipse, appears to become a straight line cutting the hydrometer scale.

8.6 To make a reading with nontransparent liquids, observe the point on the hydrometer scale to which the sample rises above its main surface, placing the eye slightly above the plane surface of the liquid. This reading requires a correction. Determine this correction for the particular hydrometer in use by observing the height above the main surface of the liquid to which the sample rises on the hydrometer scale when the hydrometer in question is immersed in a transparent liquid having a surface tension similar to that of a sample under test.

8.7 Observe the temperature of the sample to the nearest 0.25°F (0.1°C) immediately before and after the observation of the gravity, the liquid in the cylinder being thoroughly but cautiously stirred with the thermometer (Note 3), and the whole of the mercury thread being immersed. Should these temperature readings differ by more than 1°F (0.5°C), repeat the temperature and gravity observations when the temperature of the sample has become more stable. Record the mean of the thermometer reading before and after the final hydrometer reading, to the nearest 1°F, as the temperature of the test.

NOTE 3—When thermohydrometers are used, stir the sample by carefully raising and lowering the hydrometer. It is satisfactory in this case to read the thermometer scale after the hydrometer reading has been observed. Read the thermometer to the nearest 1°F (0.5°C).

TABLE 1 Available Hydrometers Scaled, Degrees API

Designation	Type	API Range, deg		Scale	
		Series Total	Each Unit	Division	Error
1H to 10H	long plain	−1 to 101	12	0.1	0.1
21H to 40H	short plain	0 to 101	6	0.1	0.2
51H to 60H	thermo	−1 to 101	12	0.1	0.1
71H to 74H	thermo	−1 to 41	12	0.1	0.1
[A]	thermo	15 to 51	8		

[A] Eight-degree range thermohydrometers are available.

9. Calculation

9.1 When gravities have been observed on opaque liquids using the procedure given in 8.6, subtract the correction from the hydrometer reading observed.

9.2 Correct all hydrometer readings to 60°F (15.56°C), using Tables 5A or Tables 5B of Guide D 1250.

TABLE 2 Limiting Conditions and Testing Temperatures

Sample Type	Gravity Limits	Initial Boiling Point Limits	Other Limits	Test Temperature
Highly volatile	lighter than 70° API			Cool to 35°F (2°C) or lower in original closed container.
Moderately volatile	heavier than 70° API	below 250°F (120°C)		Cool to 65°F (18°C) or lower in original closed container.
Moderately volatile and viscous	heavier than 70° API	below 250°F (120°C)	Viscosity too high at 65°F (18°C)	Heat to minimum temperature for sufficient fluidity.
Nonvolatile	heavier than 70° API	above 250°F (120°C)		Any temperature between 0 and 195°F (–18 and 90°C) as convenient.
Mixtures of nonpetroleum products or essentially pure hydrocarbons				60 ± 0.25°F (15.56 ± 0.1°C)

10. Report

10.1 Report the corrected hydrometer reading as degrees API (°API) or as API Gravity.

11. Precision and Bias

11.1 The precision of this test method as obtained by statistical examination of interlaboratory test results is as follows:

11.1.1 *Repeatability*—The difference between successive test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material, would in the long run, in the normal and correct operation of the test method, exceed 0.2° API only in one case in twenty.

11.1.2 *Reproducibility*—The difference between two single and independent results, obtained by different operators, working in different laboratories on identical test material, would in the long run, in the normal and correct operation of the test method, exceed 0.5° API only in one case in twenty.

NOTE 4—The precision for this method was not obtained in accordance with RR: D02-1007.

NOTE 5—This precision statement applies only to measurements made at temperatures differing from 60°F (15.56°C) by less than 18°F (10°C).

11.2 *Bias*—Bias for this method has not been determined.

12. Keywords

12.1 API gravity; crude petroleum; hydrometer; thermohydrometer; thermometer

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Cranston, RI

SOP NO. 40_0025I
ANIONS BY
ION CHROMATOGRAPHY
(METHOD EPA 300.0/SW846 9056A)

APPROVED BY:

Operations Manager	Date
QA Manager	Date
Laboratory Director	Date

Master marked in red. Controlled copies marked in green.

ANIONS BY ION CHROMATOGRAPHY (METHOD EPA 300.0/SW846 9056A)

1.0 SCOPE AND APPLICATION

1.1 This method covers the determination of Fluoride, Nitrate, Nitrite, Chloride, Ortho-Phosphate, and Sulfate

1.2 This method is applicable to drinking water, surface water, groundwater, mixed domestic and industrial waste waters, solids (after extraction) and leachate.

1.3 The method report limits for aqueous samples is as follows:

Analyte	mg/L
Fluoride	0.05
Chloride	0.50
Nitrite and Nitrate	0.02
Ortho-Phosphate	0.1
Sulfate	1.0

2.0 METHOD SUMMARY

2.1 A sample volume equal to 2 to 3 ml is introduced into an ion chromatograph. The anions are separated based on their affinities towards the stationary phase in the column. A suppressor column, packed with high capacity cation exchange resin, chemically suppresses background conductance of the eluent and, at the same time enhances the signal of the anionic species in question. The resulting signal is measured on a conductivity detector.

2.2 An extraction procedure must be performed to use this method for solids.

3.0 HEALTH AND SAFETY

3.1 Each employee has been trained and has acknowledged being trained in the safe use and handling of chemicals being used in the sample receiving area. This training has been performed according to the ESS Training SOP 80_0016 and by the Chemical Hygiene Plan, SOP No. 90_0001, in conjunction with the Safety orientation.

3.2 All sample and material handling should be done in a hood while using proper protective equipment to minimize exposure to liquid or vapor. Minimum personnel protective equipment includes the use of laboratory safety glasses, a lab coat or apron, and protective gloves.

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3.3 The MSDS for the concentrated chemicals used in the laboratory are kept on file in a central location that is available for all employees to review.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

4.1 Samples should be collected in plastic or glass bottles. All bottles must be thoroughly cleaned and rinsed with reagent water. Volume collected should be sufficient to ensure a representative sample, allow for replicate analysis (if required), and minimize waste disposal.

4.2 Sample preservation and holding times for the anions are as follows:

Analyte	Preservation	Holding Time
Fluoride, Chloride	None	28 Days
Nitrite, Nitrate, Orthophosphate	4 °C	48 Hours
Sulfate	4 °C	28 Days

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

5.1 Any substance with a retention time that is similar to or overlaps that of an analyte of interest will interfere with proper integration. Large amounts of an analyte can interfere with peak resolution of a closely eluting analyte. This is most often seen with large amounts of chloride, which will overlap with nitrite.

5.2 Method interferences may be caused by contaminants in the reagent water, reagents, glassware and instrumentation. These interferences can lead to discrete artifacts or elevated baseline in ion chromatograms.

5.3 Samples that contain particles larger than 0.45 micron and reagents with larger than 0.2 micron particle require filtration to prevent damage to columns.

6.0 EQUIPMENT/APPARATUS

6.1 **Analytical Balance** capable of accurately weighing to the nearest 0.0001 g.

6.2 **Glassware**: Class A volumetric flasks and pipettes or plastic containers as required. Samples may be stored in plastic or glass.

6.3 0.20 µm and 0.45 µm. **syringe filters**.

6.4 **Ion Chromatograph**: Analytical system complete with ion chromatograph and all required accessories including analytical columns and detectors. Lachat 8000.

6.4.1 Anion Guard Column: Protects the profiling column and is packed with the same substrate. Lachat QS-A1G 4.6x50mm Part # 28085

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6.4.2 Anion Profiling Column: An analytical column capable of separating the anions of interest. Lachat QS-A1 5.1x150mm Part# 28084

6.4.3 Anion Suppressor Column: A column which is packed with a high-capacity cation exchange resin that is capable of converting the eluent and separated anions to their respective acid forms. Lachat Chemical Suppressor

6.4.4 Detector: Temperature controlled conductivity cell. Lachat IC

6.4.5 Data System:

6.4.6 **Computer:** The Wet Chemistry laboratory has one IC system analyzing methods 300.0 and 9056A. The IC has a Gateway computer with a Windows 98 operating system. The computer systems is networked to a Windows 2000 server, which is the destination of all files. A differential back-up is performed nightly and a full back is performed each weekend using Veritas Backup Exec to tapes. As the systems acquires and stores data onto the server, the server becomes full. The data is downloaded and archived onto CDs.

6.4.7 **Software:** Lachat Omnion Chromatography Data System Version 2.0 - The software is interfaced to the electrical conductivity cells and allows the continuous acquisition and storage on machine-readable media of all chromatograms obtained throughout the duration of the instrument program.

7.0 REAGENTS AND STANDARDS

7.1 Reagents: (Note: De-gas all reagents with helium.)

7.1.1 **Reagent Water**: Deionized water with particles no larger than 0.20 µm, for preparing reagents and also for washing out the suppressor cartridge.

7.1.2 **Eluent Solution: 2.2.mM Sodium Bicarbonate/ 2.8mM SodiumCarbonate:** Dissolve 0.3696 g sodium bicarbonate and 0.5936 gm sodium carbonate in reagent water and dilute to 2L. Filter through a 0.22 µm membrane filter. De-gas by bubbling helium gas through sample for 15 min. Make fresh on the day of the analysis.

7.1.3 **Regenerant Solution - 0.25M**: Dilute 28 ml of concentrated sulfuric acid to 2L with reagent water. Make fresh on the day of the analysis.

7.2 **Standards**:

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7.2.1 **Stock Standard Solutions**: Stock standards may be purchased as certified solutions or prepared from ACS grade materials (dried at 105°C for 30 min prior to use) as listed below:

Reagent	Weight (g)	Final Volume	Conc.(mg/L)
Sodium Fluoride (NaF)	0.5526	250 ml	1000
Sodium Chloride (NaCl)	1.6484	1000 ml	1000
Sodium Nitrite ($NaNO_2$)	0.2462	500 ml	100
Potassium Nitrate (KNO_3)	0.9025	250 ml	500
Potassium Phosphate (KH_2PO_4)	1.0984	1000 ml	250
Potassium Sulfate (K_2SO_4)	0.4535	250 ml	1000

Store at 4 °C. ***Prepared monthly in DI water, except for sulfate stock (6 months) and chloride stock (1 year)***

7.2.2 **Working Standards (prepared weekly except nitrate, nitrite, and phosphate which are prepared daily)**: Prepare using stock standard as follows:

Standard ID	Cal 6	Cal5	Cal4	Cal3	Cal2	Cal1
Analyte						
NO2 mg/L	**2**	**1**	**0.5**	**0.1**	**0.05**	**0.02**
Volume (ml)	2	1	0.5	0.1	0.05	0.02
Final Vol (ml)	100	100	100	100	100	100
NO3 mg/L	**2**	**1**	**0.5**	**0.1**	**0.05**	**0.02**
Volume (ml)	0.4	0.2	0.1	0.02	0.01	1ml of 2ppm
Final Vol (ml)	100	100	100	100	100	100
F mg/L	**5**	**2-CCV**	**1-CCV**	**0.5**	**0.1**	**0.05**
Volume (ml)	0.5	0.2	0.1	0.05	0.01	1ml of 5ppm
Final Vol (ml)	100	100	100	100	100	100
Cl mg/L	**20**	**10-CCV**	**5-CCV**	**2**	**1**	**0.5**
Volume (ml)	*2*	*1*	*0.5*	*0.2*	*0.1*	*0.05*
Final Vol (ml)	100	100	100	100	100	100
SO4 mg/L	**40**	**20-CCV**	**10-CCV**	**3**	**2**	**1**
Volume (ml)	4	2	1	0.3	0.2	0.1
Final Vol (ml)	100	100	100	100	100	100
OPO4 mg/L	**8**	**4**	**2**	**1**	**0.5**	**0.2**
Volume (ml)	3.2	1.6	0.8	0.4	0.2	0.08
Final Vol (ml)	100	100	100	100	100	100

7.2.3 The **Continuing Calibration Check Standards** (CCVs) are standards prepared from the same source as that is used to generate the calibration curve. See 7.2.2 for preparation instruction. The high CCV is the same as Cal STD 5 and the low CCV is the same as Cal STD 4.

7.2.4 **Second Source Stock Standard Solutions**. Prepare second source stock standards from source different than that used to prepare primary standards (dried at 105°C for 30 min) as follows:

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Reagent	Weight (g)	Final Volume	Concentration
Sodium Fluoride (NaF)	0.0552	250 ml	100 mg/L
Sodium Chloride (NaCl)*	1.6484	1000 ml	1000 mg/L
Sodium Nitrite ($NaNO_2$)	0.1231	250 ml	100 mg/L
Potassium Nitrate (KNO_3)**	0.9025	250 ml	500 mg/L
PotassiumPhosphate(KH_2PO_4)***	1.0984	1000 ml	250 mg/L
Potassium Sulfate (K_2SO_4)	0.4535	250 ml	1000 mg/L

Expires in 3 months, except for Sulfate (6 months) and Chloride (1 year)).
* Commercially prepared at 1000 mg/L (Hach Cat #183-49)
** Commercially prepared at 100 mg/L
*** Commercially prepared at 1000 mg/L: 100 ppm stock used – see phosphate method (SOP 40.0014L).

7.2.5 The **Initial Calibration Check Standard/Blank Spike** (ICV/BS) is a mid-level standard prepared from the Intermediate Second Source Stock Standard. Prepare by pipetting 1.0 ml of each second source stock standard (§ 7.2.4) of interest into a 100 ml volumetric flask and diluting to volume with DI water. The BS contains 0.2 mg/L of Nitrate and Nitrite, 0.5 mg/L of Fluoride, 1 mg/L of Ortho-phosphate, 5 mg/L of Chloride, and 10 mg/L of Sulfate. NOTE: the ICV and BS are equivalent. ***Prepare daily.***

7.2.6 **Method Reporting Limit (MRL) Checks** are used to evaluate instrument sensitivity in an ongoing way, i.e. with each analytical batch, as an alternative to a semi-annual MDL study which the method would usually require.
MRL Check Standards will normally be the Blank Spikes for each analyte, with the exception of Fluoride, ortho-Phosphate and Sulfate, which will each require a 10% dilution of the Blank Spike.

7.2.7 **Matrix spike** (MS) is an aliquot of sample which is spiked with the standards used to prepare the blank spike. Pipette 0.1 ml of each second source stock standard (§ 7.2.4) of interest into a 10 ml aliquot of sample (or a smaller aliquot diluted to 10 ml). The concentration is equivalent to that in the blank spike.

8.0 PROCEDURE

8.1 Prepare reagents and standards.

8.2. Set up manifold. Refer to the Quickchem methods manual.

8.3 Input data system parameters: See Attachment A.

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8.4 Pump DI water through all reagent lines and check for leaks and smooth flow. Switch to reagents and all the system to equilibrate until a stable baseline is achieved.

8.5 Complete Flow Analysis Run log. See Figure 1.

8.6 Calibration:

8.6.1 Place standards in the standards rack of the autosampler. The six level curve consists of the intermediate standard (7.2.2) and the working standards (7.2.3). Input the information required by the data system, such as concentration, replicates and QC scheme.

8.6.1 Calibration is accomplished by injecting standards. The data system will then prepare a calibration curve by plotting response versus standard concentration.

8.6.2 The correlation coefficient (r) must be ≥ 0.995 for the curve to be considered valid.

8.6.3 Verify initial calibration with ICV before analysis of samples.

8.7 Retention Time Windows

8.7.1 Retention time windows are central to the identification of target compounds. Absolute retention times are used for compound identification in all IC external standard methods. Retention time windows are established to compensate for minor shifts in absolute retention times as a result of sample loadings and normal chromatographic variability. The width of the retention time window should be carefully established to minimize the occurrence of both false positive and false negative results.

8.7.2 Determining Retention Time Windows:

8.7.2.1 Before establishing retention time windows, make sure that the chromatographic system is operating reliably and that the system conditions are optimized for the target analytes in the sample matrix to be analyzed.

8.7.2.2 Make three injections of the mid-range CCV over the course of a 24 hour day.

8.7.2.3 Record the retention time in minutes for each single analyte to three decimal places. Calculate the mean and standard deviation of the three absolute retention times for each single analyte.

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8.7.2.4 If the standard deviation of the retention times for any analyte is 0.000 (i.e., no difference between the absolute retention times), then use a default standard deviation of 0.01 minutes. The width of the retention time window for each analyte is defined as ± 3 times the standard deviation of the mean absolute retention time established during the day or 0.03 minutes, whichever is greater.

8.7.2.5 Establish the center of the retention time window for each analyte by using the absolute retention time for each analyte from the mid-range CCV (Level 4 standard) at the beginning of each analytical shift. For samples run during the same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration.

8.7.2.6 Absolute retention time windows must be calculated for each analyte on each chromatographic column and instrument used.

8.7.2.7 New retention time windows must be established when a new column is installed or when major Ion chromatography maintenance has been performed.

8.8 Samples:

8.8.1 Prior to sample analysis; verify calibration with CCV and CCB. CCV and CCB must also be run after every tenth sample and at the end of the sample run.

8.8.2 Prepare solid/soil samples by weighing 10 g sample into a 150 ml beaker then adding 100 ml of DI water. Stir the sample for 10 minutes.

8.8.3 Filter all samples through a 0.45µm filter prior to analysis.

8.8.4 Place samples and appropriate QC samples in sample rack of autosampler. Input the information required by the data system, such as concentration, replicates and QC scheme.

9.0 CALCULATIONS (DATA INTERPRETATION)

9.1 Prepare a calibration curve by plotting instrument response against standard concentration. Compute sample concentration by comparing sample response with the standard curve. Multiply answer by appropriate dilution/preparation factors.

9.2 Report only those values that fall between the lowest and the highest calibration standards. Samples exceeding the highest standard should be diluted and reanalyzed.

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9.3 Report results in mg/L.

10.0 QUALITY ASSURANCE/QUALITY CONTROL

10.1 All quality control data should be maintained and available for easy reference or inspection.

10.2 Initial and Periodic Method QC Demonstrations: The procedures specified in Section 10.3.1 through 10.3.2 must be conducted as an initial demonstration of laboratory capability, prior to the analysis of any samples. Subsequent to this initial demonstration, additional evaluations of this nature should be conducted on a periodic basis, in response to changes in instrumentation or operations, and/or in response to confirmed or suspected systems, method, or operational problems.

10.3.1 Accuracy and Precision: To demonstrate initial laboratory capability, analyze a minimum of four initial calibration verification standards. The mean concentration must be within ± 10 % of the stated values.

10.3.2 Method Detection Limits for Individual Analytes: Determine in accoedance with SOP 110_0013. See Attachment B for DoD Criteria.

10.3.3 Linear Calibration Range (LCR): A new calibration curve is generated every run and the Correlation Coefficient for the range is validated.

10.3 Dilute and reanalyze samples that are more concentrated than the linear calibration limit.

10.4 The ICV must be analyzed immediately following daily calibration to verify the validity of the primary standard.

10.4.1 The ICV recovery must be within the range of 90-110%.

10.4.2 If the ICV is outside of this range, a new daily calibration must be generated.

10.5 The CCV and CCB must be analyzed prior to a sample run, after every tenth sample, and at the end of the sample run.

10.5.1 The value for the CCB must be less than ½ the MRL.

10.5.2 The CCV recovery range must be within the range of 90-110%.

10.5.3 If either the CCV or CCB are invalid, reanalyze both solutions. If the second analysis is invalid then all samples following the last acceptable CCV/CCB check solutions are invalid and must be rerun.

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10.6 Employ a minimum of one method blank per sample batch (up to 20 samples) to determine if contamination or any memory effects are occurring. The method blank must contain all the reagents and in the same volumes as used in the processing of the samples. It must be carried through the complete procedure as the sample solution used for analysis.

10.6.1 The value for the blank must be less than ½ the MRL. See Attachment B for DoD Criteria. If criteria are not achieved see section 11.0 for corrective action.

10.7 Analyze one replicate sample for every ten samples or per analytical batch, whichever is more frequent. Calculate the relative percent difference as follows:

$$RPD = \frac{D_1 - D_2}{(D_1 + D_2)/2} \times 100$$

Where: RPD = relative percent difference
D_1 = first sample value
D_2 = second sample value (replicate)

10.7.1 The control limit is +/- 20% for RPD. If the samples fall outside the control limit, see section 11.0 for corrective action.

10.8 Analyze one spike sample for every ten samples or per analytical batch, whichever is more frequent. Calculate the percent recovery as follows:

$$Recovery = \frac{SSR - SR}{SA} \times 100$$

Where: SSR = Spiked Sample Result
SR = Sample Result
SA = Spike Added to Sample

10.8.1 The Recovery control limit is 90-110% for Method 300.0 and 80-120% for Method 9056. Method 9056 also requires a Matrix Spike Duplicate with RPD≤15% (see Appendix B). If the samples fall outside the control limit, then see Section 11.0 for corrective action.

10.9 At least one BS must be analyzed for each batch of samples (up to 20 samples). The BS should be prepared and analyzed in the same manner as the samples.

10.9.1 The BS recovery range must be within the range of 90-110% (80-120% for Method 9056).

10.9.2 If the BS falls outside of this range, then see section 11.0 for corrective action.

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10.10 Any deviations from methodology or unusual observations will be noted in narrative that accompanies data.

10.11 Blanks, ICVs, and matrix spikes will be plotted in ELEMENT.

11.0 DATA VALIDATION

11.1 Data validation will be accomplished by reviewing all of the quality control parameters and assuring that they are within recommended ranges. The only exceptions made to ranges would be the following:

11.1.1 For duplicates, the RPD should be +/- 20%. However, there are cases where duplicates may not work. If this is the case, inform client in narrative concerning sample non-homogeneity.

11.1.2 For matrix spikes, the %Recovery should be +/- 10% (+/- 20% for Method 9056). If the matrix spike is outside 10%, check the Blank spike. If the Blank spike is within limits, matrix interferences are present and should be noted in the narrative.

11.1.3 For the Blank spike, the % Recovery must be within 90-110% (+/- 20% for Method 9056). If the Blank spike is outside criterion, then re-analyze. If the second analysis of the Blank spike produces results outside of criterion, then a new calibration must be performed and the associated samples re-analyzed with the following exception: ***For high Blank spike, those samples that are non-detects may be reported.***

11.1.4 Analytical batches with Method blanks above the ½ MRL will be re-prepped and re-analyzed with the following exceptions:

11.1.4.1 Samples that are at least twenty times higher than the method blank may be reported.

11.1.4.2 If the analyte is found in the method blank above the MRL but is not in any of the associated samples, no corrective action is needed.

11.1.4.3 Any results that are reported with method blank contamination must be B-flagged.

11.2 All unusual observations and method deviations will be noted in the narrative accompanying the data report presented to the client.

12.0 REFERENCES

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12.1 U.S. Environmental Protection Agency, Determination of Inorganic Anions by Ion Chromatography Method 300 Revision 2.1 August 1993.

12.2 Quickchem 8000 Training Manual, Lachat Instruments, Inc. Revised 1/12/97.

12.3 Quickchem Methods Manual, Lachat Instruments, Inc., Revised 7/9/1997.

12.4 Quickchem Method 10-510-00-1-A, Anions by Ion Chromatography, Revised 7/9/97.

12.5 NELAC Quality Systems Chapter 5, June 2003

12.6 DoD Quality Systems Manual, Final Version 4.1, April 2009

13.0 POLLUTION PREVENTION and WASTE MANAGEMENT

13.1 ESS Laboratory's policies and pollution prevention and waste management are covered in SOP 90_0002, Hazardous Waste Contingency and Emergency Response Plan. All employees are trained in the requirements of the SOP.

14.0 DEFINITIONS

14.1 **Calibration Blank (CB)** -- A volume of reagent water fortified with the same matrix as the calibration standards, but without the analytes.

14.2 **Calibration Standard (CAL)** -- A solution prepared from the primary dilution standard solution or stock standard solutions. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

14.3 **Blank spike (BS)** -- An aliquot of reagent water or other blank matrices to which known quantities of the method analytes are added in the laboratory. The BS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.

14.4 **Matrix spike (MS)** -- An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The MS is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS corrected for background concentrations.

14.5 **Method Blank (MB)** -- An aliquot of reagent water or other blank matrices that are treated exactly as a sample including exposure to all glassware, equipment, and reagents that are used with other samples. The MB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

14.6 **Material Safety Data Sheet (MSDS)** -- Written information provided by vendors concerning a chemical's toxicity, health hazards, physical properties, fire, and reactivity data including storage, spill, and handling precautions.

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14.7 **Method Detection Limit (MDL)** -- The minimum concentration of an analyte that can be identified measured and reported with 99% confidence that the analyte concentration is greater than zero.

14.8 Also see DoD QSM V4.1, Appendix B – Glossary, which may be found in Network Directory Q:\DOD\ DoDV4.1.

15.0 METHOD PERFORMANCE

15.1 Precision and Accuracy data must be generated by all employees before performing this analysis on client samples. The data is generated by analyzing a method blank and four blank spike samples. Acceptance criteria are 90-110% Recovery and %RSD of $\leq$ 20%.

15.2 The precision and accuracy data in Table 1 were developed using a reagent water matrix. Values are in mg/L.

16.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

Table 1. Typical Precision and Accuracy data generate

Analyte	Date	True value	Average	%Recovery	%RSD
Sulfate	9/29/07	10	10.06	101	0.5
Chloride	10/24/07	10	10.32	103	0.3
Fluoride	10/24/07	1	1.05	105	0.4

17.0 PERSONNEL QUALIFICATIONS

17.1 Analysts who perform this analysis must have a working knowledge of quantitative and qualitative analysis, instrumental methods of analysis, chemical laboratory methods, and equipment.

17.2 All analysts, before performing any analysis, participate in the ESS Laboratory training program (SOP80_0016). The training process consists of reading the Standard Operating Procedure, gaining instruction on the procedure from an experienced analyst, and performing the initial demonstration of capability.

18.0 TROUBLESHOOTING

18.1 **Daily Maintenance**: Analyst should clean any spills that occur on or around the instrument at the end of each shift. When the column is not in use, cap ends of column and store in refrigerator.

18.2 See laboratory supervisor or operations manager for all other maintenance problems.

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18.3 Record all maintenance in the instrument's maintenance logbook.

19.0 DATA MANAGEMENT AND RECORDS

19.1 **Data Management** - ESS Laboratory utilizes the Promium Element LIMS system as part of its Data Management system. Client sample information is entered into ELEMENT LIMS and analyses are assigned to each sample. The LIMS allows EPA hold times, minimum batch QC requirements, and QC criteria to be assigned to each analysis. Standards can be entered and assigned to QC samples through the LIMS. Once analysis has been performed, data is imported using DataTool avoiding manual errors. In conjunction with Crystal Reports, the ELEMENT system allows for a wide variety of reporting formats.

19.2 **Records** – The specific retention periods required in the NELAC Standards, EPA-CFR and state and local statutes are followed or exceeded. At a minimum, data records are retained for five years from last use (10 years for drinking water). If there is a question about whether a record should be retained or disposed because no specific requirement could be found, the record is retained until such time as a retention period is specified. Records are stored in specified-labeled locations and are easily retrievable. All raw data associated with testing is also retained including; computer printouts, chromatograms, review forms, and logbooks.

20.0 ATTACHMENTS

20.1 Figure 1 - Flow Analysis Run log

20.2 Attachment A - Lachat data system parameters.

20.3 Attachment B – DoD QSM V4.1 Method Quality Objectives, Table F-11.

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Figure 1: Flow Analysis Run Log

Date: _____
Analyst: _____
Batch: _____ Analyte: _____
BS/MS std: _____ File ID: _____
Rgnt: _____ ID: _____ Exp. _____
Rgnt: _____ ID: _____ Exp. _____
Rgnt: _____ ID: _____ Exp. _____
Rgnt: _____ ID: _____ Exp.

Date: _____
Analyst: _____
Batch: _____ Analyte: _____
BS/MS std: _____ File ID: _____
Rgnt: _____ ID: _____ Exp. _____
Rgnt: _____ ID: _____ Exp. _____
Rgnt: _____ ID: _____ Exp. _____
Rgnt: _____ ID: _____ Exp.

#	SAMPLE ID	DIL	Spk vol	#	SAMPLE ID	DIL	Spk vol
#	Method blank/CCB			#	Method blank/CCB		
#	Std 1-			#	Std 1-		
#	Std 2-			#	Std 2-		
#	Std 3-			#	Std 3-		
#	Std 4-			#	Std 4-		
#	Std 5-			#	Std 5-		
#	Std 6-			#	Std 6-		
#	ICV			#	ICV		
#	CCV			#	CCV		
1				1			
2				2			
3				3			
4				4			
5				5			
6				6			
7				7			
8				8			
9				9			
10				10			
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Table F-11. Common Anions Analysis (Method 9056)					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see Section C.1.f).	NA.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification	At initial set-up and verified quarterly. If a laboratory uses multiple instruments for a given method, the LOD must be verified on each.	The apparent signal to noise ratio must be at least 3 and the results must meet all method requirements for analyte identification.	If the LOD verification fails, the laboratory must: 1) Repeat the detection limit determination and LOD verification at a higher concentration; or 2) Perform and pass two consecutive LOD verifications at a higher concentration. The LOD is set at the higher concentration.	NA.	Samples may not be analyzed without a valid LOD.
LOQ establishment and verification	At initial setup: 1) Verify LOQ; and 2) Determine precision and bias at the LOQ. Subsequently, verify LOQ quarterly. If a laboratory uses multiple instruments for a given method, the LOQ must be verified on each. See Box D-14.	1) The LOQ and associated precision and bias must meet client requirements and must be reported; or 2) In the absence of client requirements, must meet control limits of the LCS. See Box D-14.	If the LOQ verification fails, the laboratory must either establish a higher LOQ or modify method to meet the client-required precision and bias.	NA.	Samples may not be analyzed without a valid LOQ.
Retention time (RT) window width calculated for each analyte	After method set-up and after major maintenance (e.g., column change).	RT width is ± 3 times standard deviation for each analyte RT over a 24-hour period.	NA.	NA.	

Table F-11. Common Anions Analysis (Method 9056) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial calibration (ICAL) for all analytes (minimum three standards and one calibration blank)	ICAL prior to sample analysis.	$r \geq 0.995$.	Correct problem, then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed.
Initial calibration verification (ICV) (second source)	Once after each ICAL, prior to beginning a sample run.	All analytes within ± 10% of true value and retention times within appropriate windows.	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
Retention time window position establishment for each analyte	Once per multipoint calibration.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	
Midrange continuing calibration verification (CCV)	After every 10 field samples and at the end of the analysis sequence.	All project analytes within established retention time windows. Within ± 10% of true value.	Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed. Retention time windows are updated per the method.
Method blank	One per preparatory batch.	No analytes detected > ½ RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results (see Box D-1).	Correct problem, then see criteria in Box D-1. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table F-11. Common Anions Analysis (Method 9056) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
LCS containing all analytes to be reported	One per preparatory batch.	Laboratory in-house limits not to exceed ± 20%. Control limits may be not greater than ± 3 times the standard deviation of the mean LCS recovery. See Box D-3.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available (see full explanation in Appendix G).	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch per matrix (see Box D-7).	For matrix evaluation, use laboratory in-house LCS limits (not to exceed ± 20%).	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix spike duplicate (MSD)	One per preparatory batch per matrix (see Box D-7).	For matrix evaluation, use laboratory in-house LCS limits (not to exceed ± 20%). RPD ≤ 15% (between MS and MSD).	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Sample duplicate (replicate)	One per every 10 samples.	%D ≤ 10% (between sample and sample duplicate).	Correct problem and reanalyze sample and duplicate.	Apply J-flag if sample cannot be rerun or reanalysis does not correct problem.	The data shall be evaluated to determine the source of difference.
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

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ESS Laboratory
Division of Thielsch Engineering
Cranston, RI

SOP NO. 50_3510C

SEPARATORY FUNNEL LIQUID - LIQUID EXTRACTION

(SW-846 METHOD 3510C)

REVIEWED BY:

Operations Manager	Date 1/9/7
QA Officer	Date 1/9/07
Laboratory Director	Date 1/9/07

MASTER

Master copy marked red. Controlled copies marked green.

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Separatory Funnel Liquid - Liquid Extraction
(SW-846 Method 3510C)

1.0 SCOPE AND APPLICATION

1.1 This method describes a procedure for isolating organic compounds from aqueous samples by separatory funnel extraction. It is applicable to the following determinative methods:

Pesticide 8081	PCB 8082
DRO 8015BM	Pest/PCB (608)
PAHs by 8270C	TPH 8100M/CT ETPH
Low Level PAH & PCP	EPH MCP CAM IVB

2.0 METHOD SUMMARY

2.1 A measured volume of sample, usually 1 liter, at a specified pH is serially extracted with methylene chloride using a separatory funnel. The extract is collected, dried (de-watered), concentrated (if necessary), and, as necessary, exchanged into a solvent compatible with the clean-up or determinative method to be used.

3.0 HEALTH AND SAFETY

3.1 The toxicity and carcinogenicity of each reagent used in this method has not been precisely defined. However, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available.

3.2 Each employee has been trained and has acknowledged being trained in the safe use and handling of chemicals being used in the laboratory. This training has been performed according to the ESS Training SOP 80_0016 and by the Chemical Hygiene Plan, SOP No. 90_0001, in conjunction with the Safety orientation.

3.3 All sample and material handling should be done in a hood while using proper protective equipment to minimize exposure to liquid or vapor. Minimum personnel protective equipment includes the use of laboratory safety glasses, a lab coat or apron, and protective gloves.

3.4 The MSDS for the concentrated chemicals used in the laboratory are kept on file in a central location that is available for all employees to view.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

4.1 Sample containers will be borosilicate glass with Teflon lined screw caps, typically 1.0L amber borosilicate bottles. They will be purchased pre-cleaned and ready for use. Sample containers are stored in the storage area adjacent to the sample receiving area. ESS Laboratory retains limited control over client supplied samples

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and some samples may arrive in substandard containers. Any unusual observations will be noted in the final report.

4.2 The analytical methods utilizing this method for the extraction of aqueous samples will dictate the preservative and the quantity of that preservative to be used in conjunction with the holding time.

Method	Preservative	Extraction Hold Time
Pesticide 8081	None	7 Days
PCB 8082	None	7 Days
Pest/PCB (608)	5-7	7 Days
Pest/PCB (608)	< 5, >7	48 Hours
DRO 8015M	pH<2 with HCl	7 Days
TPH 8100M	pH<2 with HCl	7 Days
PAH/PCP	None	7 Days
EPH MCP CAM IVB	pH<2 with HCl	14 Days

4.3 In addition to chemical preservation, samples are stored at 4 ± 2°C until extraction. The samples are located in the walk-in cooler located in the sample storage room. All samples must be bar-coded in and out of the refrigerator, see SOP 70_0006.

4.4 All samples will be handled as potentially hazardous materials. As a minimal precaution, safety glasses, laboratory coat, and impermeable gloves will be worn when handling samples.

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

5.1 Glassware, including separatory funnels, must be carefully cleaned prior to each use in order to remove potential contaminants. Refer to the organic prep glassware cleaning SOP 50_0001.

5.2 The use of method blanks to determine that solvents and reagents used in extraction are free of interfering contaminants is incorporated into each analytical batch.

5.3 In some cases an emulsion will form when the solvent is added to the aqueous sample. These emulsions can cause low surrogate and spike recoveries.

5.4 The following cleanup methods can be used to remove certain interferences: (See Cleanup SOPs for procedures)

5.4.1 PCB Analysis

5.4.1.1 Sulfuric Acid Cleanup to remove most organic interferences.

5.4.1.2 Copper Cleanup to remove sulfur.

5.4.2 Pesticide Analysis

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5.4.2.1 Copper Cleanup

5.4.2.2 Carbo-Prep Sep-Pak Cartridge Cleanup

5.4.2.3 GPC Cleanup

Note: Never use Sulfuric Acid to clean a pesticide extract. Certain pesticides will breakdown.

5.4.3 DRO/TPH analysis:

5.4.3.1 Sulfuric Acid Clean up to remove naturally occurring organics. Only applied if requested. Must be noted in project narrative.

5.4.4 PAH Analysis

5.4.4.1 GPC Cleanup

5.4.4.2 Silica gel cleanup for PAHs.

6.0 EQUIPMENT/APPARATUS

6.1 **Separatory funnel**: 2 liter Teflon with Teflon stopcock.

6.2 **Turbovap II** Concentration workstation (Zymark)

6.2.1 Concentration tubes, 200 ml with 1.0 ml (part#: 45817)

6.3 **Boiling chips**: Teflon 10/40 mesh silicon.

6.4 **Vials**: 2 ml, glass with Teflon lined screw-caps or crimp tops.

6.5 **pH Indicator Paper**: ColorpHast® wide range.

6.6 **Erlenmeyer flasks**: 250 ml, 500 ml

6.7 **Syringe**: 1.0 ml glass bore Hamilton syringe.

6.8 **Graduated cylinder**: 1 liter.

7.0 REAGENTS AND STANDARDS

7.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy

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of the determination. Reagents should be stored in glass or Teflon to prevent the leaching of contaminants from plastic containers.

7.2 All reagents are logged and tracked according to the Organic Prep Lab Reagent Tracking SOP 50_0006.

7.3 **Organic-free reagent water**: All references to water in this method refer to organic-free reagent water. The water is obtained from the ESS DI water system. This water is checked for target organics on a regular basis by the analysis of method blanks.

7.4 **Sodium hydroxide solution** (10N), NaOH: Dissolve 40 g NaOH in organic-free reagent water and dilute to 100 ml.

7.5 **Sodium sulfate** (granular, anhydrous), Na_2SO_4: The anhydrous sodium sulfate **must** be muffled in a furnace at 400°C for 4 hours. A method blank prepared with the muffled sodium sulfate must be analyzed to demonstrate that there are no interferences from the sodium sulfate.

7.6 **Sulfuric acid** H_2SO_4, 18N: ACS reagent grade 36N. Dilute 1:1 in DI H_2O.

7.7 **Sodium thiosulfate** $Na_2S_2O_3$, ACS reagent grade.

7.8 **Extraction/exchange solvents**

7.8.1 **Methylene chloride**, CH_2Cl_2: Pesticide quality or equivalent.

7.8.2 **Hexane**, C_6H_{14}: Pesticide quality or equivalent.

7.9 **Surrogate and Spike Solutions:** The solutions are prepared according to instructions in the appropriate determinative method. The instrument laboratory must check all new spiking solution before they are used in an extraction. The concentrations of the analytes in the spiking solution **must be within 90-110%** of the true value. All of the quality control data for spiking solutions is stored in the instrumentation laboratory.

8.0 PROCEDURE

8.1 Setup

8.1.1 All Separatory Funnels must be thoroughly cleaned according to SOP 50_0001.

8.1.1.1 Wash separatory funnels, stopcocks, and caps with soap and water. Thoroughly rinse off the soap.

8.1.1.2 Place the separatory funnels on the ring stands located in the ventilation hood. Rinse once with Methanol to remove all traces of

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water.

8.1.1.3 Use a methylene chloride squirt bottle to rinse the inside of each funnel, stopcock, and cap. Place a beaker on the bottom of the funnel to catch the waste solvent. Discard waste solvent in the waste solvent satellite located under the ventilation hood.

8.1.1.4 Allow the funnel to completely air dry.

8.1.1.5 Turn the stopcock in the off position.

8.2 **Batching of samples:** All samples are set up as an analytical batch. The following must be true for samples to be considered a batch:

8.2.1 A batch consists of a group of 20 or less field samples of a similar matrix.

8.2.2 A preparation batch is defined as samples that are prepared together by the same person, or group of people, using the same equipment/glassware with the same method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in limited continuous sequential time periods, usually not to exceed 24 hours (same operators).

8.3 **Retrieve samples for analysis:**

8.3.1 Review the ESS Laboratory Organic Prep Back Log that was generated that morning.

8.3.2 Select 20 or fewer samples that require the 3510C extraction for a particular determinative method. (Any of those listed in 1.1) If there are more than twenty samples, select samples according to the following criteria

8.3.2.1 Sample which have hold times that are going to expire that day or the next should be analyzed first.

8.3.2.2 If hold times are not an issue then analyze samples according to their due date.

8.3.3 The samples are stored in the ESS Laboratory walk-in cooler; the shelves are labeled with the ESS Laboratory project ID numbers.

8.3.4 Log out all samples from the walk-in cooler. (Refer to the Bar-code SOP 70_0006)

8.3.5 The batch of samples is transferred from the walk-in cooler, using a cart, to the Organic Prep Lab. The samples are then placed in a ventilation hood in the organic prep lab.

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8.3.6 All samples in the batch are logged into the Organic Prep logbook (Attachment A). The following information is documented:

8.3.6.1 The ESS project numbers. This includes all projects that are being extracted in the batch.

8.3.6.2 Prep Date

8.3.6.3 The Batch ID

Example: BA50201

Where: B = Identified it as a Batch
A = Identifies the month (A for January, B for February, etc.)
5 = Identifies the year (last digit of the year)
02 = Identifies the date (this batch was created on Jan. 2nd)
01 = First batch prepared for that day

8.3.6.4 The analysis to be performed is checked off.

8.3.6.5 The analytical matrix is recorded as aqueous.

8.3.6.6 The extraction method is 3510C

8.3.6.7 The working standard IDs of the surrogates and spikes (this means all standards used) used are written into the blank spaces provided in this logbook..

8.3.6.8 The ESS IDs of all samples to be extracted are documented. This includes QC samples.

8.3.6.9 The remainder is completed as the extraction is performed.

8.3.7 The batch is set up with the following QC standards:

8.3.7.1 **METHOD BLANK**: 1L of lab DI water, measured with a 1L graduated cylinder, is poured into a clean separatory funnel. The water will be spiked with the appropriate surrogate solution, extracted, concentrated and analyzed like a sample. This is a quality control sample known to be free of any target analytes or contaminants. It is analyzed to verify that samples were not contaminated during extraction or analysis.

8.3.7.2 **BLANK SPIKE/BLANK SPIKE DUPLICATE:** 1L of lab DI water, measured with a 1L graduated cylinder, is poured into a separatory funnel. The water will be spiked with the appropriate surrogate, spiking solution, extracted, concentrated and analyzed like a sample. This is a quality control sample that contains known

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concentrations of target analytes and is free from any sample matrix interference. It is analyzed to verify that under optimum conditions the target analytes can be accurately and precisely recovered.

8.3.7.3 **Matrix Spike/Matrix Spike Duplicate:** One sample is randomly selected to be the sample, matrix spike and matrix spike duplicate. In some cases the client will specify which sample is to be used as the matrix spike and matrix spike duplicate sample. These are quality control samples to which known concentrations of target analytes are added. They are analyzed to document the effect of the sample matrix on the recovery of the target analytes.

8.4 **Extraction when the entire sample bottle is used: (If only a fraction of the sample is used go to section 8.5)**

NOTE: ***All spiking (surrogate or blank/matrix spike) must be witnessed by a second analyst. The Spike witness is to record their initials in the provided column.***

8.4.1 Allow the sample to come to room temperature.

8.4.2 Mark the meniscus of the sample bottle. ***If samples are received with an appreciable amount of sediment contained in the sample bottle, then allow the sediment to settle and decant off the liquid portion into a graduated cylinder to determine the volume. Avoid transferring the sediment into the graduated cylinder.*** **See section 8.5 for using sample fractions.**

8.4.3 Randomly select a sample for the matrix spike and matrix spike duplicate. In some cases the client will specify which sample is to be used.

8.4.4 **Check the initial pH of the sample** with wide-range pH paper. Transfer a small volume of sample with a clean glass rod or glass disposable pipette to the pH paper. Record pH in logbook. If the sample was properly preserved proceed to 8.4.6; if not then go to section 8.4.5.

8.4.5 Adjust the pH, if necessary, to the pH indicated in the following table. Use 1:1 Sulfuric acid or 10N Sodium hydroxide. The amount of acid or base added to the sample should not result in a significant change (<1%) in the volume of sample extract.

Determinative Method	Initial Extraction pH	Secondary pH
8081 Pesticides	5-9	none
8082 PCB	5-9	none
DRO/8100/8015 TPH	As received	none
PAHs by 8270	>11	N/A
Low Level PAH	>11	N/A
Low Level PCP/EPH	≤2	N/A

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8.4.6 Samples must be tested to determine if residual chlorine is present. This may be accomplished by transferring a small volume of sample with a clean glass rod or glass disposable pipette to starch iodide paper (do not dip the test paper in the sample). A blue color is indicative of residual chlorine. Record a positive result in the logbook. Discharge residual chlorine by adding 3 ml of sodium thiosulfate per gallon (or 0.008%). This step is to be carried out immediately following step 8.4.2.

8.4.7 Transfer the entire contents of the sample bottle into the separatory funnel.

8.4.8 With a calibrated syringe add the appropriate surrogate solution directly to the separatory funnel containing the method blank, blank spike, blank spike duplicate, samples, and matrix spike/matrix spike duplicate.

8.4.8.1 For pesticides 8081A/608 and PCB 8082/608 add 0.5 ml of the pesticide/PCB surrogate. For Low Level PCBs, add 0.1 ml of PCB/Pesticide surrogate.

8.4.8.2 For PAH 8270C add 1.0 ml of the PAH surrogate.

8.4.8.3 For TPH 8100M or CT-ETPH, add 1.0 ml of the TPH surrogate.

8.4.8.4 For DRO 8015B, add 1 ml of TPH Surrogate.

8.4.8.5 For Low Level PAH (including CT-RCP), add 0.25 ml of LLPAH surrogate. Spiked samples also require 0.2 ml of the LLPAH and PCP Matrix Spike solution; then proceed as described in § 8.4.5.

8.4.8.6 For Low Level PCP, add 0.25 ml of LLPAH surrogate. Spiked samples additionally require 0.2 ml of LLPAH and PCP Matrix Spike solution; then proceed as described in § 8.4.5.

8.4.9 1 ml of spike (0.5 ml for Pesticides and PCBs) or 0.1 ml for LLPCB's is added to the blank spike, matrix spike and matrix spike duplicate sample. (See determinative method for details on the spike standard solution.

8.4.10 Label the separatory funnel with the ESS lab ID #. The ID is written on a small piece of paper tape that is placed on the funnel.

8.4.11 Rinse the sample bottle with methylene chloride by pouring about 60ml of methylene chloride into the bottle in the ventilation hood. Swirl the methylene chloride in the bottle to remove any remaining sample. Do not spill any methylene chloride.

8.4.12 Quantitatively pour the methylene chloride into the separatory funnel containing the aqueous sample.

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8.4.13 Fill the bottle to the meniscus with tap water. Pour the water into a 1000 ml graduated cylinder. Measure the volume (ml) of water in the graduated cylinder. This volume is the volume of sample extracted. Record the exact sample volume in the initial volume column of the organic prep logbook.

8.4.14 Continue with section 8.6.

8.5 **Extracting a fraction of the sample from a single bottle:**

8.5.1 Allow the sample to come to room temperature.

8.5.2 Mix contents of bottle by gentle shaking bottle. ***Caution: When sediment is present in sample bottle do not disturb sediment layer, see section 8.4.2.***

8.5.3 Immediately remove the cap from the sample bottle to vent.

8.5.4 Pour the amount of sample to be used for the 3510C extraction into a clean glass graduated cylinder. (The graduated cylinder must be pre-rinsed with methylene chloride.

8.5.5 Measure the exact amount of sample in the graduated cylinder in ml. Record the volume of sample in the Organic prep log.

8.5.6 **Check the initial pH of the sample** with the wide-range pH paper. Transfer a small volume of sample with a clean glass rod or glass transfer pipette to the pH paper. If the sample was preserved properly proceed to 8.5.8; if not then make a note of the initial pH in the Organic prep logbook.

8.5.7 Adjust the pH if necessary to the pH indicated in the table in Section 8.4.5.

8.5.8 Test for residual chlorine and treat, if necessary, as described in Section 8.4.6.

8.5.9 Transfer an aliquot of the sample to a graduated cylinder. Pour the entire contents of the graduated cylinder into the separatory funnel.

8.5.10 1 ml of spike (0.5 ml for Pesticides and PCBs) is added to the blank spike, matrix spike and matrix spike duplicate sample. (Refer to the determinative method for details on the spike standard solution.)

8.5.11 Label the separatory funnel with the ESS lab ID #. The ID is written on a small piece of paper tape that is placed on the funnel.

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8.5.12 With a 1.0 ml calibrated syringe add 1.0 ml of the appropriate surrogate solution directly to the sample in the separatory funnel.

8.5.12.1 For pesticides 8081A/608 and PCBs 8082/608 add 0.5 ml of

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the pesticide/PCB surrogate. For Low Level PCBs, add 0.1 ml of PCB/Pesticide surrogate.

8.5.12.2 For PAH 8270C add 1.0ml of the PAH surrogate.

8.5.12.3 For TPH 8100M or CTETPH add 1.0 ml of the TPH surrogate.

8.5.12.4 For DRO 8015B and DRO 4.1.25 add 1 ml of TPH surrogate.

8.5.12.5 For Low Level PAH (including State of Connecticut protocols), add 0.25 ml of LLPAH surrogate. Spiked samples additionally require 1.0 ml of the LLPAH and PCP Matrix Spike solution; then proceed as described in Paragraph 8.4.5.

8.5.12.6 For Low Level PCP, add 0.25 ml of LLPAH surrogate. Spiked samples additionally require 1.0 ml of the LLPAH and PCP Matrix Spike solution; then proceed as described in Paragraph 8.4.5.

8.5.13 Rinse the graduated cylinder with methylene chloride by pouring about 60ml of methylene chloride into the cylinder in the ventilation hood. Swirl the methylene chloride in the cylinder. Do not spill any methylene chloride!

8.5.14 Quantitatively pour the methylene chloride into the separatory funnel containing the aqueous sample.

NOTE: For volumes of 500 ml or less, DI water is to be added to the sample after the surrogate spiking solution (and matrix spike when appropriate) has been added. The final volume is to be approximately 1000 ml after the addition of the DI water. The initial volume of the sample is recorded in prep logbook **not** the volume after dilution.

8.6 Seal and shake the separatory funnel vigorously for 1 to 2 minutes with frequent venting to release excess pressure.

NOTE: Methylene chloride creates excessive pressure very rapidly; therefore, initial venting should be done immediately after the separatory funnel has been sealed and shaken once. Venting of the separatory funnel should be into a hood to avoid exposure of the analyst to solvent vapors.

8.7 Allow the organic layer to separate from the water phase for a minimum of 10 minutes. If the emulsion interface between layers is more than one-third the size of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample and may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods.

8.8 Place a glass funnel into the top of the concentrator tube. Use a small piece of glass wool to plug the funnel. Pour approximately 100g of dry sodium sulfate into the

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funnel. The solvent extract is drained through the sodium sulfate and collected in the concentrator tube.

8.9 Add another 60 ml aliquot of methylene chloride to the separatory funnel. Repeat the extraction two more times. (Sections 8.6 through 8.8)

8.10 After the final extraction, rinse the sodium sulfate with the solvent used for extraction. Transfer the ESS Lab ID to the Turbovap concentrator tube. The sodium sulfate and glass wool in the funnel is disposed of in the laboratory solid waste satellite drum.

8.10.1 **Semi-volatile extractions**: Semi-volatiles are performed by SOP 50_3520C (except for PAHs and Phthalates by method 8270C).

8.11 **Concentration:** Turbovap Technique.

8.11.1 The Turbovap technique uses the Zymark Turbovap II workstation. This system uses a warm water bath and a continuous flow of nitrogen to concentrate samples. Turn on the Turbovap using the button on the lower left-hand side. Fill water bath to the indicated fill mark and set bath temperature to 40 $\pm$ 2°C. The temperature of the bath must be checked with a calibrated thermometer each day of use and recorded on Attachment B.

8.11.2 The Turbovap can be programmed to stop after a certain time, manually or using an optical sensor. When the sensor is used, the Turbovap will stop ("beep") when the sample extract reaches 0.75 ml. For dirty samples, the "time" option should not be used due to false sensor readings. Adjust the gas pressure using the knob on the left-hand side (behind the power button). Pull knob out and turn clockwise to increase pressure and counterclockwise to decrease pressure. The pressure should be set at 15-20 psi. For samples that are 200ml or greater, start at a lower pressure (6psi) then increase when sample has evaporated below 200ml mark on concentrator tube. This will eliminate the risk of sample loss due to splashing.

8.11.3 Place concentrator tubes with extract into the Turbovap. Adjust bath temperature and gas flow and start by pressing the appropriate cell number (1-6). The Turbovap will "beep" when extract has reached 0.75 ml or when the preset time has expired.

8.11.4 When samples are being analyzed for Pesticides or PCBs, a solvent exchange is also required. When the original extract has been evaporated to approximately 1.0 ml, add approximately 30 ml of hexane and evaporate to 0.75 ml. Bring the final volume up to the calibrated 5 ml mark using hexane. The extract is now ready for analysis. ***The laboratory uses a "go-by" to adjust the extract to a final volume greater than 1 ml. The "go-by" is prepared by pipetting Methylene chloride into a 15 ml vial with a 5 ml class A pipette, prepared daily to avoid changes from evaporation.***

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8.11.5 When PAH, TPH, DRO, EPH and low-level pesticides/PCBs are being concentrated, the final extract volume should be 1.0 ml. After the Turbovap has finished concentrating to 0.75 ml transfer the extract to a 2ml target vial. Bring the final volume up to 1.0 ml with methylene chloride (Hexane for Pesticides/PCBs) using a syringe. The ESS Lab ID is transcribed onto the target vial.

8.11.6 When Low Level PAH or Low Level PCP are being concentrated, the final extract volume should be 0.25 ml.

8.11.7 If a clean-up procedure is needed, refer to the clean-up SOP for further instructions.

8.11.8 The extract is now ready to be analyzed for the target analytes using the appropriate determinative technique(s). All extracts must be analyzed within 40 days.

9.0 CALCULATIONS (DATA INTERPRETATION)

9.1 See Determinative Methods.

10.0 QUALITY ASSURANCE/ QUALITY CONTROL

10.1 The equipment in contact with the sample and reagents is interference-free. This is accomplished through the analysis of a liquid matrix method blank (e.g., DI Water). Each time samples are extracted, and when there is a change in reagents, a method blank must be extracted and analyzed for the compounds of interest. The method blank should be carried through all stages of the sample preparation and measurement.

10.2 **Accuracy and Precision**: All laboratory personnel must demonstrate initial proficiency for each sample preparation method/matrix that he or she performs prior to independently performing analysis on real samples. This is accomplished by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The initial proficiency results become part of each employee's training file.

10.2.1 QC Sample Preparation:

10.2.1.1 Spiking Solution: Four QC samples must be prepared from a spiking solution with the analytes of interest. The spiking solution must be made using standards **prepared independently from those used for calibration**. The samples must be prepared at a concentration that would result in data falling within the middle of the calibration curve.. Prep: The samples are prepared in a clean matrix. In most cases, this initial demonstration will simply be a matter of preparing four blank spikes with a batch of samples. (See the determinative method for preparation of this spiking solution.)

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10.2.2 Sample Analysis: The four QC samples must be analyzed within the criteria of the method being evaluated. The QC samples must be handled in exactly the same manner as actual samples.

10.2.3 Accuracy Calculation: Accuracy is defined as the closeness of agreement between an observed value and an accepted reference value. Each of the four spiked samples will be calculated for percent recovery. The average of the percent recovery values is the accuracy result.

10.2.4 Precision Calculation: Precision is defined as the agreement of a set of replicate measurements without assumption of knowledge of the true value. Precision is estimated by the relative standard deviation (RSD) of the four QC samples.

%RSD = (s / x) 100 %

Where:
s = Standard Deviation of a finite number of values. On a scientific calculator, use the **σxn-1** key.

$\bar{x}$ = The average of the four QC sample % recoveries.

10.2.5 Reporting Accuracy and Precision: Accuracy and Precision data should be presented with the following minimum info:

Matrix: Prep Method:
Clean-up Method: Analysis Method:
Date Extracted: Date Analyzed:

Sample Prepared by: If Applicable
Sample Fractionated by: If Applicable Sample Analyzed by:
Accuracy: Precision:

Parameter	*% Rec. QC 1*	*% Rec. QC 2*	*% Rec. QC 3*	*% Rec. QC 4*	*Average Recovery*	*Standard Deviation*	*%RSD*

10.2.6 Interpretation of Results: Refer to the determinative method SOP for specific criteria. If any of the accuracy and precision results do not fall within the established criteria, then re-prep and reanalyze all QC samples only for those analytes that did not meet criteria.

10.3 Standard quality assurance practices are performed with this method. Field duplicates should be collected to validate the precision of the sampling procedures. A matrix spike/matrix spike duplicate, or matrix spike and duplicate sample analysis, and a blank spike/blank spike duplicate should be prepared and analyzed with each batch of samples prepared by this procedure, unless the determinative method provides other guidance.

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10.4 For DoD work, the target analytes, as they are defined in the DoD QSM, Version 4.2, Appendices DoD-B and DoD-D, must be spiked in the QC samples (LCS, MS, MSD). Additionally, all target analytes must be included in the spike mixture over a two year period.

10.5 Surrogate and spike standards are added to samples according to the appropriate determinative method.

10.6 Corrective action for out of criteria results are found in the determinative SOP.

11.0 DATA VALIDATION

11.1 Any unusual observations are noted in the prep logbook.

11.2 The laboratory supervisor routinely reviews all logbooks.

11.3 All non-compliance requires a corrective action report. This report must be included with the raw data. Refer to the quality control section of the determinative method.

12.0 REFERENCES

12.1 SW846 Method 3510C, Separatory Funnel Liquid-Liquid Extraction, Third Edition, Update III.

12.2 NELAC/NELAP Standards, Chapter 5, June 2003.

12.3 DoD QSM, Version 4.2, October 2010.

12.4 ESS Laboratory Policy and Procedure.

13.0 POLLUTION PREVENTION and WASTE MANAGEMENT

13.1 ESS Laboratory's policies on pollution prevention and waste management are covered in SOP 90_0002, Hazardous Waste Contingency and Emergency Response Plan. All employees are trained in the requirements of the SOP.

14.0 DEFINITIONS

14.1 Internal Standard - A pure analyte(s) added to a sample, extract, or standard solution in known amount(s) and used to measure the relative responses of other method analytes and surrogates that are components of the same sample or solution. The internal standard must be an analyte that is not a sample component.

14.2 Surrogate Analyte - A pure analyte(s), which is extremely unlikely to be found in any sample, and which is added to a sample aliquot in known amount(s) before extraction or other processing and is measured with the same procedures used to measure other sample components. The purpose of the SA is to monitor method

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performance with each sample.

14.3 Laboratory Duplicate – A second aliquot of a sample taken in the laboratory and analyzed separately with identical procedures as the sample. Analyses of the lab duplicate and sample indicates precision associated with laboratory procedures, but not with sample collection, preservation, or storage procedures.

14.4 Field Duplicates - Two separate samples collected at the same time and place under identical circumstances and treated exactly the same throughout field and laboratory procedures. Analyses of the sample and its field duplicate give a measure of the precision associated with sample collection, preservation and storage, as well as with laboratory procedures.

14.5 Method Blank - An aliquot of reagent water or other blank matrix that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The Method Blank is used to determine if method analytes or other interferences are present in the laboratory environment, reagents, or the apparatus.

14.6 Field Blank (FB) - An aliquot of reagent water or other blank matrix that is placed in a sample container in the laboratory and treated as a sample in all respects, including shipment to the sampling site, exposure to sampling site conditions and equipment, storage, preservation, and all analytical procedures. The purpose of the FB is to determine if method analytes or other interferences are present in the field environment.

14.7 Blank Spike - An aliquot of reagent water or other blank matrix to which known quantities of the method analytes are added in the laboratory. The BS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.

14.8 Matrix Spike (MS) - An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The MS is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS corrected for background concentrations.

14.9 Stock Standard Solution - A concentrated solution containing one or more method analytes prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source.

14.10 Calibration Standard (CAL) - A solution prepared from the primary dilution standard solution or stock standard solutions and the internal standards and surrogate analytes. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

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14.11 Quality Control Sample - A solution of method analytes of known concentrations which is used to fortify an aliquot of BS or sample matrix. The quality control sample (known as second source standard) is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check laboratory performance with externally prepared test materials.

15.0 METHOD PERFORMANCE

15.1 See determinative methods for method performance information.

16.0 PERSONNEL QUALIFICATIONS

16.1 BA/BS Chemistry, Natural & Applied Science or equivalent experience.

16.2 All analysts, before performing any analysis, participate in the ESS Laboratory training program (SOP80_0016). The training process consists of reading the Standard Operating Procedure, gaining instruction on the procedure from an experienced analyst, and performing the initial demonstration of capability.

17.0 TROUBLESHOOTING

See determinative method.

18.0 DATA MANAGEMENT AND RECORDS

18.1 **Data Management** - ESS Laboratory utilizes the Promium Element LIMS system as part of its Data Management system. Client sample information is entered into ELEMENT LIMS and analyses are assigned to each sample. The LIMS allows EPA hold times, minimum batch QC requirements, and QC criteria to be assigned to each analysis. Standards can be entered and assigned to QC samples through the LIMS. Once analysis has been performed, data is imported using DataTool avoiding manual errors. In conjunction with Crystal Reports, the ELEMENT system allows for a wide variety of reporting formats.

18.2 **Records** –The specific retention periods required in the NELAC Standards, EPA-CFR and state and local statutes are followed or exceeded. At a minimum, data records are retained for five years from last use (10 years for drinking water). If there is a question about whether a record should be retained or disposed because no specific requirement could be found, the record is retained until such time as a retention period is specified. Records are stored in specified-labeled locations and are easily retrievable. All raw data associated with testing is also retained including; computer printouts, chromatograms, review forms, and logbooks.

19.0 ATTACHMENTS (including TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA)

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19.1 Attachment A – Organic Preparation Logbook

19.2 Attachment B – Turbovap Temperature Log

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Attachment A ESS Organic Preparation Logbook

Project #:_____ Surrogate ID# Matrix Spike ID# Analytical Matrix:_____

Prep Date:_____ A_____ D_____ Extraction Time:

Batch ID:_____ B_____ E_____ Start:_____

Extraction Method:_____ C_____ F_____ Finish:_____

Split Extraction* ☐

* Half of the final extract volume (0.5ml) is exchanged into 5ml 5ml hexane and transferred as Vol 1. The other half (0.5ml CH_2Cl_2) is transferred as Volume 2.

ESS ID	Vol(ml)/ Wt.(g)	Surrogate (ul or ml)	Matrix Spike (ul or ml)	Final Extract Vol (ml) Hex/CH_2Cl_2	Transfer Vol #1 (ml) Hex/CH_2Cl_2	Transfer Vol #2 (ml) Hex/CH_2Cl_2	Transfer Date	Bath Temp (C)	pH	Discard bottle**	Comments	1st Rvw Init.	Witness Init.	2nd Rvw Init.

Analysis Performed

- PCB ☐
- B/N SVOA ☐
- SVOA ☐
- LL PAH ☐
- PEST ☐
- TPH/GC ☐
- BIS-2 ☐
- PAH ☐

Acid Washed: Y / N H_2SO_4 ID#	Cu Cleaned: Y / N Cu ID#	Florisiled: Y / N Lot#	Silica Column/Carbon prep: Y / N Lot #

CH_2Cl_2 lot #_____ NaOH ID#_____

Prepared By:_____ Glasswool:_____ Method #(s):_____ Hexane lot#_____ Na_2SO_4 ID#_____

**Check off column if entire sample used and bottle discarded.

Acetone lot#_____

Control #50.0001-0701A **BATCH ID/Test:**_____ **BATCH ID/Test:**_____ **Page**_____

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TurboVap Temperature LOG

Location _____ Number _____

Thermometer Reading Range 40 +/- 2 °C

Thermometer serial # _____

Correction Factor _____ Month / Year _____

	TurboVap 1	TurboVap 2	TurboVap 3	TurboVap 4			
Day	**TEMP°C**	**TEMP°C**	**TEMP°C**	**TEMP°C**	**Time**	**Initials**	**Comments**
1							
2							
3							
4							
5							
6							
7							
8							
9							
10							
11							
12							
13							
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ESS Laboratory
Division of Thielsch Engineering
Cranston, RI

SOP NO. 50_3540C
SOXHLET EXTRACTION
(SW-846 METHOD 3540C)

REVIEWED BY:

Operations Manager	Date 5/1/07
QA Officer	Date 5/1/07
Laboratory Director	Date 5/1/07

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SOXHLET EXTRACTION
(SW-846 METHOD 3540C)

1.0 SCOPE AND APPLICATION

1.1 Method 3540 is a procedure for extracting nonvolatile and semi-volatile organic compounds from solids such as soils, sludges, and wastes. The Soxhlet extraction process ensures intimate contact of the sample matrix with the extraction solvent.

1.2 This method is applicable to the isolation and concentration of water-insoluble and slightly water-soluble organics in preparation for a variety of chromatographic procedures.

2.0 SUMMARY OF METHOD

2.1 The solid sample is mixed with anhydrous sodium sulfate, placed in an extraction thimble or between two plugs of glass wool, and extracted using an appropriate solvent in a Soxhlet extractor. The extract is then dried, concentrated (if necessary), and, as necessary, exchanged into a solvent compatible with the cleanup or determinative step being employed.

3.0 HEALTH AND SAFETY

3.1 Each employee has been trained and has acknowledged being trained in the safe use and handling of chemicals. This training has been performed according to the ESS Training SOP 80_0016 and by the Chemical Hygiene Plan, SOP No. 90_0001, in conjunction with the Safety orientation.

3.2 All sample and material handling should be done in a hood while using proper protective equipment to minimize exposure to liquid or vapor. Minimum personnel protective equipment includes the use of laboratory safety glasses, a lab coat or apron, and protective gloves.

3.3 Stock solutions and reagents used for sample preservation are used in a hood.

3.4 The MSDS for the concentrated chemicals used in the laboratory are kept on file in a central location that is available for all employees to review.

4.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

4.1 Sample containers will be borosilicate glass with Teflon lined screw caps, typically 8 oz. jars. They will be purchased pre-cleaned and ready for use.

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4.2 Samples are extracted as soon as possible. When immediate extraction is not possible, samples are to be stored at 4°C for up to 14 days from the sampling date.

5.0 INTERFERENCES

5.1 Soxhlet extractors, flasks and other glassware, must be carefully cleaned prior to each use in order to remove potential contaminants. See SOP 50_0001.

5.2 Solvents and reagents used in extraction are quality control checked with method blank to ensure they are acceptable for analytical use.

5.3 Phthalate esters will contaminate samples if plastic processing apparatus are used. Care should be taken to avoid any contact with plastic materials.

5.4 If necessary, Florisil, sulfuric acid, and/or sulfur cleanup procedures may be employed. In such cases, proceed with SOP No. 50_3620B, SOP No. 50_3665A, and/or SOP No. 50_3660B.

6.0 EQUIPMENT/APPARATUS AND MATERIALS

6.1 **Soxhlet extractor**: 45-50-mm i.d., with 500-ml round-bottom flask.

6.2 **Filter funnel**: 90-mm Pyrex glass with 10-mm sodium sulfate stopped with muffled glass wool.

6.3 **Turbovap II** Concentration workstation

6.3.1 Concentration tubes, 200 ml with 1.0 ml marking (part#: 45817)

6.4 **Boiling chips**: Solvent extracted, approximately 10/40 mesh Teflon.

6.5 **Vials**: Glass, 2-ml capacity, with Teflon lined screw caps or crimp tops.

6.6 **Glass or paper thimble** or **glass wool**: Contaminant free.

6.7 **Heating mantle**: Rheostat controlled.

6.8 **Hamilton Syringe**: 5-ml.

6.9 **Apparatus for determining percent moisture**:

6.9.1 Oven: Drying, VWR® Maintained at 105° C

6.9.2 Desiccator, portable

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6.9.3 Disposable aluminum weigh pans

6.10 **Pasteur glass pipettes**: 1.0 ml, disposable.

6.11 **Beakers**: 400 ml.

6.12 **Erlenmeyer flasks**, wide-mouthed: 500 ml

6.13 **Calibrated Balance**: Top loading, OHAUS® and Precision Standard TS-400D

6.14 **Glass scintillation vials**: 20 ml, with Teflon lined screw caps.

6.15 **Spatula**: Stainless steel or Teflon.

6.16 1.0-ml glass bore **Hamilton Syringe**.

7.0 REAGENTS AND STANDARDS

7.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 Organic-free reagent water. All references to water in this method refer to organic-free reagent water, as defined in Chapter One, SW-846.

7.3 Sodium sulfate (granular, anhydrous), Na_2SO_4. The anhydrous sodium sulfate must be muffled in a furnace (~4 hours) or solvent rinsed (i.e.: 4 hours with a soxhlet extraction apparatus) prior to use as a drying agent on sample extracts. Purify by heating at 400° C for 2 hours in a shallow tray, or by pre-cleaning the sodium sulfate with methylene chloride. If the sodium sulfate is pre-cleaned with methylene chloride, a method blank must be analyzed, demonstrating that there is no interference from the sodium sulfate.

7.4 Extraction Solvents

7.4.1 Soil/sediment and aqueous sludge samples shall be extracted using the following solvent system:

7.4.1.1 Acetone/Hexane(1:1-v/v), $CH_3COCH_3/CH_3(CH_2)_4CH_3$. Pesticide quality or equivalent.

7.4.2 Other samples shall be extracted using the following:

7.4.2.1 Methylene chloride, CH_2Cl_2. Pesticide quality or equivalent.

7.5 Exchange Solvents

7.5.1 Hexane, C_6H_{14}. Pesticide quality or equivalent.

8.0 PROCEDURE

8.1 **Batching of samples:** All samples are set up as an analytical batch. The following must be true for samples to be considered a batch:

8.1.1 A batch consists of a group of 20 or less field samples of a similar matrix.

8.1.2 A preparation batch is defined as samples that are prepared together by the same person, or group of people, using the same equipment/glassware with the same method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in limited continuous sequential time periods, usually not to exceed one analytical shift.

8.2 Retrieve samples for analysis:

8.2.1 Review the ESS Organic Prep Backlog that was generated that morning.

8.2.2 Select 20 or less samples that require the 3540C extraction for a particular determinative method. If there are more than twenty samples select samples according to the following criteria

8.2.2.1 Sample which have hold times that are going to expire that day or the next should be analyzed first.

8.2.2.2 If hold times are not an issue then analyze samples according to their due date.

8.2.3 The samples are stored in the ESS walk-in cooler on the shelves labeled with project numbers.

8.2.4 Log out all samples from the walk-in cooler. (Refer to the Sample Storage SOP 10_0002)

8.2.5 The batch of samples is transferred from the walk-in cooler, to the Organic Prep Lab.

8.2.6 All samples in the batch are logged into the Organic Prep logbook. The following information is documented:

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8.2.6.1 The ESS project numbers. This includes all projects that are being extracted in the batch.

8.2.6.2 Prep Date

8.2.6.3 The Blank ID which is also the batch ID. Assigned through the Element LIMs system.

8.2.6.4 The analysis to be performed is checked off.

8.2.6.5 The analytical matrix is written as soil.

8.2.6.6 The extraction method is 3540C

8.2.6.7 The ESS working standard IDs of the surrogates and spikes used are written into the blank spaces provided.

8.2.6.8 The ESS IDs of all samples to be extracted are documented. This includes QC samples.

8.2.6.9 The remainder is completed as the extraction is performed.

8.2.7 The batch is set up with the following QC standards:

8.2.7.1 **Method Blank**: An equivalent amount (to sample weight) of anhydrous Sodium Sulfate will be spiked with the appropriate surrogate, and placed in an extraction thimble, extracted, concentrated and analyzed like a sample. This quality control sample is free of any target analytes or contaminants. It is analyzed to verify that samples were not contaminated during extraction or analysis. If target analytes or other contaminants are present in the method blank then the entire batch must be re-extracted.

8.2.7.2 **Blank Spike and Blank Spike Duplicate:** An equivalent amount (to sample weight) of anhydrous Sodium Sulfate will be spiked with the appropriate surrogate and spike solution, placed in an extraction thimble, extracted, concentrated, and analyzed like a sample. This quality control sample contains known concentrations of target analytes and is free from any sample matrix interference. It is analyzed to verify that under optimum conditions, without any sample matrix interference, the target analytes can be accurately recovered. If the recoveries of these target analytes are outside of established control limits then the entire batch must be re-extracted.

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8.2.7.3 **Matrix Spike/Matrix Spike Duplicate:** One sample is randomly selected to be the sample, matrix spike and matrix spike duplicate. In some cases the client will specify which sample is to be used as the matrix spike and matrix spike duplicate sample. These are quality control samples, which contains known concentrations of target analytes in the sample matrix. They are analyzed to document the effect of the sample matrix on the recovery of the target analytes.

8.3 Fill out the preparation log with all appropriate information prior to starting the extraction See Attachment 2.

8.3.1 The lot numbers and reagent IDs for all reagents used for extraction.

8.3.2 Document all QC and samples to be analyzed.

8.4 Sample Handling

8.4.1 Sediment/soil samples - Soil samples received for organic preparation typically have the consistency of coarse dry sand. In order to obtain a representative subsample for analysis, ESS Laboratory is guided by the subsampling techniques described in Attachment G of SOP 10.0001 (Sample Receiving), which calls for homogenization by stirring. Accordingly, the sample is thoroughly mixed, either in the original container, or if it should be necessary, by transferring to a larger clean glass container, and stirring with a clean glass rod. After mixing, weigh out an appropriate amount of sample (to the nearest 0.1 g) into a previously identified Soxhlet thimble. This is performed by discrete transfers from different parts of the sample container, avoiding any artifacts, such as rocks and twigs, and taking care to use all of the material transferred, in the analysis. In the event that the consistency differs from that described above, then Subsampling Attachment G, referred to above, must be consulted for further guidance. Record this weight in the logbook.

8.5 Determination of percent solids: When sample results are to be calculated on a dry weight basis, a second portion of sample is weighed at the same time as the portion used for analytical determination.

8.5.1 WARNING: The drying oven should be contained in a hood or vented. Significant laboratory contamination may result from drying a heavily contaminated sample.

8.5.2 Immediately after weighing the sample for extraction, weigh 10 g of the sample into a tared aluminum pan. Dry this aliquot at least 4 Hours at

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105°C. Allow to cool in a desiccator before weighing. Calculate the % solids as follows:

$$\% \text{ Solids} = \frac{\text{Dry weight - pan weight}}{\text{wet weight - pan weight}} \times 100$$

Record the weights and percent solids in the Percent Solids Logbook (see Attachment 3).

8.6 **Sample Preparation for extraction**: Blend the indicated amount (see below) of solid sample with sufficient anhydrous sodium sulfate to facilitate extraction and place in an extraction thimble. Transfer small aliquots from different parts of the sample container until the required amount has been transferred (do not remove from the beaker any excess transferred sample).

Standard sample weights, in grams, are as follows:

Pesticides	20
PCBs (Soil)	20
PCBs (Concrete)	10
SVOA	15
LLPAH	15
TPH/DRO	20
EPH	25

8.7 The extraction thimble must drain freely for the duration of the extraction period. A glass wool plug above and below the sample in the Soxhlet extractor is an acceptable alternative for the thimble. Add 1.0 ml of the surrogate standard spiking solution onto the sample. See appropriate determinative method for spiking solutions. For the sample in each analytical batch selected for spiking, add the appropriate volume of the matrix-spiking standard.

8.8 Place 300 ml of the extraction solvent (Section 5.3) into a 500-ml round-bottom flask containing one or two clean boiling chips. Attach the round bottom flask to the extractor. Place a heating mantle under the flask and adjust the mantle so that the flask fits tightly. Set the heating mantle to setting #4. At this temperature, the solvent should gently boil. Once the heating mantle comes to temperature, adjust the cycle time to 4 - 6 cycles/hr. The cycle time is the time it takes the thimble to fill with solvent and then completely drain. The thimble should start draining every 10-15 minutes to achieve the 4-6 cycles/hr. If the extractor drains in less than 10 minutes, reduce the heating mantle temperature. If the extractor takes greater than 15 minutes to drain, then increase the heating mantle temperature. Once the cycle time is set, extract the sample for 18 ± 2 hours.

8.9 Allow the extract to cool after the extraction is complete.

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8.10 **Concentration Procedure**: Turbovap Technique.

8.10.1 The Turbovap technique uses the Zymark Turbovap II concentration workstation. This system uses a warm water bath and a continuous flow of nitrogen to concentrate samples. Turn on the Turbovap using the button on the lower left-hand side. Fill water bath to the indicated fill mark and set bath temperature to 40 $\pm$ 2°C. The temperature of the bath must be checked with a calibrated thermometer each day of use and recorded in the TurboVap Temp Log (Attachment 4).

8.10.2 The Turbovap can be programmed to stop after a certain time, manually, or using an optical sensor. When the sensor is used, the Turbovap will stop ("beep") when the sample extract reaches 0.75 ml. For dirty samples, the "time" option should be used due to false sensor readings. Adjust the gas pressure using the knob on the left hand side (behind the power button). Pull knob out and turn clockwise to increase pressure and counterclockwise to decrease pressure. The pressure should be set at 10-15 psi. For samples that are 200ml or greater, start at a lower pressure then increase when sample has evaporated below 200ml mark on concentrator tube. This will eliminate the risk of sample loss due to splashing.

8.10.3 To dry the extract, place a glass funnel into the top of the concentrator tube. Use a small piece of glass wool to plug the funnel. Pour approximately 100g of dry sodium sulfate into the funnel.

8.10.4 Quantitatively pour the sample into the funnel. The sample should filter through the sodium sulfate and glass wool. All water in the sample should be absorbed by the sodium sulfate. No water should pass into the tube. Refilter if water is present in the tube.

8.10.5 To ensure that the entire sample has been transferred into the concentration tube, rinse the flask containing the original extract with approximately 5 ml of appropriate solvent (methylene chloride or in the case of PCB analysis, hexane). Collect all rinsate in the concentration tube. Only fill concentrator tube to 200 ml. If more extract remains, evaporate a portion of the extract in the tube then decant the rest of the extract. Rinse flask with approximately 5 ml appropriate solvent and decant tube. Transfer the ESS Lab ID to the concentrator tube.

8.10.6 Remove the funnel containing sodium sulfate. The sodium sulfate and glass wool in the funnel is disposed of in the laboratory solid waste container.

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8.10.7 Place concentrator tubes with extract into the Turbovap. Adjust bath temperature and gas flow and start by pressing the appropriate cell number (1-6). The Turbovap will "beep" when extract has reached 0.75 ml or when the preset time has expired.

8.10.8 When samples are being analyzed for EPH, a solvent exchange is required. When the original extract has been evaporated to 0.75 ml, add approximately 30 ml of hexane and evaporate to 0.75 ml. For EPHs, transfer the extract to a calibrated target vial. Bring the final volume up to the calibrated 1.0 ml mark using hexane. The extract is now ready for the fractionation procedure. Refer to the EPH SOP 50_EPH2.

8.10.9 When samples are being analyzed for Pesticides or PCBs, a solvent exchange is also required. When the original extract has been evaporated to approximately 1.0 ml, add approximately 30 ml of hexane and evaporate to 0.75 ml. Bring the final volume up to the calibrated 10 ml mark using hexane. The extract is now ready for analysis. ***The laboratory uses a "go-by" to adjust the extract to a final volume greater than 1 ml. The "go-by" is prepared by pipetting Methylene chloride into a 15 ml vial with a 10 ml class A pipette, prepared daily to avoid changes from evaporation.***

8.10.10 PAH, EPH, TPH, DRO, and low-level PCB's are concentrated by the Turbovap procedure to 0.75 ml, and the extract transferred to a 2 ml target vial ***The laboratory uses a "go-by" to adjust the extract to a final volume of 1 ml. The meniscus height is adjusted to the height of the meniscus in the go-by vial. The "go-by" is prepared by transferring 1ml of hexane with a calibrated 1ml Hamilton syringe to a 2ml target vial and capped. The go-by vial is prepared daily to avoid changes from evaporation.*** . The ESS Lab ID is transcribed onto the sample target vial.

8.11 The extract is now ready to be analyzed for the target analytes using the appropriate determinative technique(s). All extracts must be analyzed within 40 days.

9.0 CALCULATIONS

9.1 Not applicable: See determinative method.

10.0 QUALITY CONTROL

10.1 Before processing any samples, the analyst should demonstrate that all parts of the equipment in contact with the sample and reagents are interference-free. This is accomplished through the analysis of a solid matrix method blank (see section 8.2.7.1). Each time samples are extracted, and when there is a change in reagents, a method blank (use same amount as sample weight) must be extracted

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and analyzed for the compounds of interest. The method blank should be carried through all stages of the sample preparation, clean-up, and measurement.

10.2 Standard quality assurance practices should be used with this method. Field duplicates should be collected to validate the precision of the sampling procedures. A matrix spike/matrix spike duplicate, or matrix spike/duplicate sample analysis, and a laboratory control sample should be prepared and analyzed with each batch of samples prepared by this procedure, unless the determinative method provides other guidance. Use same amount as sample weight.

10.3 Surrogate and spike standards should be added to samples according to the appropriate determinative method.

11.0 DATA VALIDATION

11.1 Refer to the determinative methods for performance data.

11.2 Any unusual observations are noted in the prep log.

11.3 All logbooks are routinely reviewed by the laboratory supervisor.

11.4 All non-compliances require a corrective action report. This report must be included with the raw data. Refer to the quality control of the determinative method.

12.0 REFERENCES

12.1 U. S. EPA 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule," 1997.

12.2 Rohrbough, W. G.; et al. Reagent Chemicals, American Chemical Society Specifications, 7th ed.; American Chemical Society: Washington, D.C., 1986.

12.3 SW846 Method 3540C, (12/96) Soxhlet Extraction, third edition. Update III, 12.3.1 Volume 1B, chap. 4, sec. 4.2.1

12.4 ESS Laboratory Policies and Procedure.

12.5 NELAC Standards, Chapter 5, June, 2003

12.6 DoD QSM, Final Revision 4.2, October 2010

13.0 POLLUTION PREVENTION and WASTE MANAGEMENT

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13.1 ESS Laboratory's policies on pollution prevention and waste management are covered in SOP 90_0002, Hazardous Waste Contingency and Emergency Response Plan. All employees are trained in the requirements of the SOP.

14.0 METHOD PERFORMANCE

14.1 Precision and Accuracy data must be generated by all employees before performing this analysis on client samples. The data is generated by analyzing a method blank and four blank spike samples. Acceptance criteria are 80-120% Recovery and %RSD of $\leq$ 20%. Five (5) sporadic marginal exceedances are allowed.

14.2 The precision and accuracy data in Table 1 were obtained using the SOP. Values are in ug/L.

15.0 DEFINITIONS

15.1 **Accuracy**: The closeness of agreement between an observed value and an accepted reference value. When applied to a set of observed values, accuracy will be a combination of a random component and of a common systematic error (or bias) component.

15.2 **Batch**: A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 20, then each group of 20 samples or less will all be handled as a separate batch.

15.3 **Bias**: The deviation due to matrix effects of the measured value (x_s - x_u) from a known spiked amount, where x_s is the spiked sample and x_u is the un-spiked sample. Bias can be assessed by comparing a measured value to an accepted reference value in a sample of known concentration or by determining the recovery of a known amount of contaminant spiked into a sample (matrix spike).

15.4 **Control Sample**: A QC sample introduced into a process to monitor the performance of the system.

15.5 **Equipment Blank**: A sample of analyte-free media which has been used to rinse the sampling equipment. It is collected after completion of decontamination and prior to sampling. This blank is useful in documenting adequate decontamination of sampling equipment.

15.6 **Method Reporting Limit**: The lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine

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laboratory operating conditions. The MRL is generally 5 to 10 times the MDL. ESS Laboratory sets the MRL to the lowest non-zero standard in the calibration curve or higher.

15.7 **Field Duplicates**: Independent samples which are collected as close as possible to the same point in space and time. They are two separate samples taken from the same source, stored in separate containers, and analyzed independently. These duplicates are useful in documenting the precision of the sampling process.

15.8 **Blank Spike (BS)**: A known matrix spiked with compound(s) representative of the target analytes. This is used to document laboratory performance.

15.9 **Matrix**: The component or substrate (e.g., surface water, drinking water) which contains the analyte of interest.

15.10 **Matrix Duplicate**: An intralaboratory split sample which is used to document the precision of a method in a given sample matrix.

15.11 **Matrix Spike**: An aliquot of sample spiked with a known concentration of target analyte(s). The spiking occurs prior to sample preparation and analysis. A matrix spike is used to document the bias of a method in a given sample matrix.

15.12 **Matrix Spike Duplicates**: Intralaboratory split samples spiked with identical concentrations of target analyte(s). The spiking occurs prior to sample preparation and analysis. They are used to document the precision and bias of a method in a given sample matrix.

15.13 **Method Blank**: An analyte-free matrix (in this case, anhydrous sodium sulfate) to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank should be carried through the complete sample preparation and analytical procedure. The method blank is used to document contamination resulting from the analytical process.

15.14 **Method Detection Limit (MDL)**: The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix type containing the analyte. See SOP 110_0013 for further explanation.

15.15 **Organic-Free Reagent Water**: For volatiles, all references to water in the method refer to water in which an interferant is not observed at the method

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detection limit of the compounds of interest. A water purification system is usedto generate organic-free deionized water.

15.16 **Records**: Include all logbooks, papers, machine readable materials, or other documentary materials, regardless of physical form or characteristics.

15.17 **Surrogate**: An organic compound which is similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which is not normally found in environmental samples.

15.18 **Trip Blank**: A sample of analyte-free media taken from the laboratory to the sampling site and returned to the laboratory unopened. A trip blank is used to document contamination attributable to shipping and field handling procedures. This type of blank is useful in documenting contamination of volatile organics samples.

16.0 PERSONNEL QUALIFICATIONS

16.1 BA/BS Chemistry, Natural & Applied Science or equivalent experience.

16.2 All analysts, before performing any analysis, participate in the ESS Laboratory training program (SOP80_0016). The training process consists of reading the Standard Operating Procedure, gaining instruction on the procedure from an experienced analyst, and performing the initial demonstration of capability.

17.0 TROUBLESHOOTING

See Determinative method.

18.0 DATA MANAGEMENT AND RECORDS

18.1 **Data Management** - ESS Laboratory's utilizes the Promium Element LIMS system as part of its Data Management system. Client sample information is entered into ELEMENT LIMS and analyses are assigned to each sample. The LIMS allows EPA hold times, minimum batch QC requirements, and QC criteria to be assigned to each analysis. Standards can be entered and assigned to QC samples through the LIMS. Once analysis has been performed, data is imported using DataTool avoiding manual errors. In conjunction with Crystal Reports, the ELEMENT system allows for a wide variety of reporting formats.

18.2 **Records** – The specific retention periods required in the NELAC Standards, EPA-CFR and state and local statutes are followed or exceeded. At a minimum, data records are retained for five years from last use (10 years for drinking water). If there is a question about whether a record should be

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retained or disposed because no specific requirement could be found, the record is retained until such time as a retention period is specified. Records are stored in specified-labeled locations and are easily retrievable. All raw data associated with testing is also retained including; computer printouts, chromatograms, review forms, and logbooks.

19.0 ATTACHMENTS

Attachment 1 – PCB Congeners in Fish Tissue

Attachment 2 – Organic Prep Logbook

Attachment 3 – Solids Logbook

Attachment 4– TurboVap Temperature logbook

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Attachment I

Project Specific SOP Modification

PCB Congeners in Fish Tissue

Project ID: PCB Congeners in Fish Tissue/Mollusk

SOP(s) Modified: PCB Congener SOP 50_3540 and 60_8082CONG

Modification Objective: Modified sample preparation and extraction procedure for PCB Congeners in Fish Tissue/Mollusk.

Specific SOP Modifications: SOP 3540 Soxhlet Extraction. Samples are extracted as stated in the 50_3540 SOP with the following exceptions.

Method Summary

The laboratory receives fish fillets or mollusk tissue at 4 ± 2°c. Tissue may be composited for extraction. Each grab or composite sample is assigned a unique ESS sample identification number. The tissue is kept frozen until ready for extraction. Prior to extraction, the tissue is processed with a tissue grinder for physical reduction and homogenization. The homogenized samples can be split for PCB, Pesticide, Metals and any other analysis required. Tissue samples for PCB and Pesticide analysis are extracted by EPA SW846 method 3540 using 5 g of tissue. EPA SW846 method 8082, using an acid cleanup, is used to measure PCB congeners by capillary column gas chromatography with electron capture detectors. Detection limits for PCB congeners are approximately 5 ppb. Tissue sample results are reported in ug/Kg wet weight.

Sample Collection, Preservation and Handling: Fish samples will be received filleted and mollusk samples will be received shucked. Fish and mollusk tissue will be placed in glass jars containing Teflon lined screw caps. From time sampled to time received by lab, tissue must be kept at 4°C. Upon receipt, the sample is frozen. Samples must be extracted within 14 days of date sampled.

Interferences

A florisil cleanup is necessary when analyzing Pesticides in fish tissue due to interferences from fatty acids. Acid washing samples to be analyzed for PCB's will eliminate most organic interferences.

Equipment/Apparatus and Materials

Tissue grinder for physical reduction and homogenization: "Cuisinart" BDH-2.

Spiking Solutions:

Surrogate: 1ml of the Congener surrogate at 50 ug/ml is added to samples and QC.

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Spike : 1ml of the congener spike at 50 ug/ml is added to blank spikes and MS/MSDs.

Homogenization: Prior to extraction, all of the tissue supplied for a particular sample is removed from the freezer and thawed at room temperature for about an hour, or until it is somewhat pliable. The homogenizer will not work if tissue is frozen solid. In the homogenizer the tissue gets cut up into small fragments less than 1mm. The homogenized tissue is split up among various clean glass jars with Teflon lined screw caps. (One jar for each analysis performed on the tissue) The PCB congener extraction is performed with 5.0 grams of sample. If an MS/MSD is to be performed on a sample, a total of 15 grams of sample is used.

Sample Preparation for extraction: In a clean 100 ml beaker, blend 5.0 g of the homogenized tissue sample with approximately 5 g of anhydrous sodium sulfate. Continue adding anhydrous sodium sulfate and stirring the sample until a granular dry texture is obtained. A glass wool plug is placed in the base of the soxhlet extractor. Place the entire dried sample with anhydrous sodium sulfate above the glass wool. Add 1.0 ml of the surrogate standard spiking solution onto the sample. For the sample in the analytical batch selected for spiking, add 1.0 ml of the matrix-spiking standard.

Sample Concentration: When low level pesticides and PCBs are being concentrated, the final extract volume should be 1.0 ml. After the Turbovap has finished concentrating samples in hexane to 0.75 ml, bring the extract to 1.0 ml using a calibrated syringe and quantitatively transfer to a 2.0-ml target vial. The ESS Lab ID is transcribed onto the target vial.

The extract is now ready to be analyzed for the target analytes using the appropriate determinative technique(s). All extracts must be analyzed within 40 days.

Batching:
Each twenty samples must be digested with a method blank, blank spike, matrix spike sample, and duplicate sample.

Data Reporting: All results are reported to the Method Reporting Limit of 5 ug/Kg.

The following is reported with each batch of samples:

- Method blank
- Blank Spike
- Sample results
- Matrix spike result
- Duplicate sample result

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Attachment 2

ESS Organic Preparation Logbook

Project #:_____ Surrogate ID# Matrix Spike ID# Analytical Matrix:_____

Prep Date:_____ A_____ D_____ Extraction Time:

Batch ID:_____ B_____ E_____ Start:_____

Extraction Method:_____ C_____ F_____ Finish:_____

Split Extraction* ☐

* Half of the final extract volume (0.5ml) is exchanged into 5ml 5ml hexane and transferred as Vol 1. The other half (0.5ml CH_2Cl_2) is transferred as Volume 2.

ESS ID	Vol(ml)/ Wt.(g)	Surrogate (ul or ml)	Matrix Spike (ul or ml)	Final Extract Vol (ml) Hex/CH_2Cl_2	Transfer Vol #1 (ml) Hex/CH_2Cl_2	Transfer Vol #2 (ml) Hex/CH_2Cl_2	Transfer Date	Bath Temp (C)	pH	Discard bottle**	Comments	1st Rvw Init.	Witness Init.	2nd Rvw Init.

Analysis Performed

- PCB ☐
- B/N SVOA ☐
- SVOA ☐
- LL PAH ☐
- PEST ☐
- TPH/GC ☐
- BIS-2 ☐
- PAH ☐

Acid Washed: Y / N H_2SO_4 ID#	Cu Cleaned: Y / N Cu ID#	Florisiled: Y / N Lot#	Silica Column/Carbon prep: Y / N Lot #

CH_2Cl_2 lot #_____ NaOH ID#_____

Prepared By:_____ Glasswool:_____ Method #(s):_____ Hexane lot#_____ Na_2SO_4 ID#_____

**Check off column if entire sample used and bottle discarded. Acetone lot#_____

Control #50.0001-0701A BATCH ID/Test:_____ BATCH ID/Test:_____ Page_____

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Percent Solids Logbook

Date	Lab ID	Pan WT. (g)	Wet WT. (g)	Dry WT. (g)	Percent Solid	Wet wt. Init.	Dry wt./1st Rvw Init.	2nd Rvw Init.

Criteria: Dup RPD ≤ 20% Control #50.0006-0203A Page_____

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Attachment 4

ESS Laboratory
Division of Thielsch Engineering, Inc.

TurboVAP Temperature LOG

Location : Organic Prep Lab

Thermometer Reading Range 45 ± 2 °C

Thermometer serial # _____

Correction Factor _____ Month / Year 09/2002

TuboVap #1 #2 #3 #4

DAY	TEMP°C	TEMP°C	TEMP°C	TEMP°C	TIME	Initials	Comments
1							
2							
3							
4							
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Control# 10_0004at3
Attachment B
SOP 50_3540C

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ESS Laboratory
Division of Thielsch Engineering
Cranston, RI

SOP NO. 50_3541
AUTOMATED SOXHLET EXTRACTION
(SW-846 METHOD 3541)

REVIEWED BY: _____ 8/14/07
Operations Manager Date

_____ 8/14/07
QA Officer Date

_____ 8/13/07
Laboratory Director Date

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AUTOMATED SOXHLET EXTRACTION

1.0 SCOPE AND APPLICATION

1.1 Method 3541 is a procedure for extracting nonvolatile and semi-volatile organic compounds from solids such as soils, sludges, and wastes. The Soxhlet extraction process ensures intimate contact of the sample matrix with the extraction solvent.

1.2 There are two differences between this extraction method and method 3540. In the initial extraction stage of method 3541, the sample loaded extraction thimble is immersed into the boiling solvent. This ensures very rapid intimate contact between the specimen and solvent and rapid extraction of the organic analytes. In the second stage, the solvent is evaporated to a level just below the thimble and is rinse-extracted as in method 3540. A third stage can then be applied to evaporate the solvent, as would occur in the TurboVap Technique concentration step in method 3540.

2.0 SUMMARY OF METHOD

2.1 The solid sample is mixed with anhydrous sodium sulfate, placed in an extraction thimble, with one plug of glass wool on bottom, and extracted using an appropriate solvent in a Soxtherm Automated Extractor. The extract is then dried and concentrated (if necessary), and, as necessary, exchanged into a solvent compatible with the cleanup or determinative step being employed.

3.0 HEALTH AND SAFETY

3.1 Each employee has been trained and has acknowledged being trained in the safe use and handling of chemicals. This training has been performed according to the ESS Training SOP 80_0016 and by the Chemical Hygiene Plan, SOP No. 90_0001, in conjunction with the Safety orientation.

3.2 All sample and material handling should be done in a hood while using proper protective equipment to minimize exposure to liquid or vapor. Minimum personnel protective equipment includes the use of laboratory safety glasses, a lab coat or apron, and protective gloves.

3.3 The MSDS for the concentrated chemicals used in the laboratory are kept on file in a central location that is available for all employees to review.

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4.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

4.1 Sample containers will be borosilicate glass with Teflon lined screw caps, typically 250-ml wide mouth amber borosilicate bottles. They will be purchased pre-cleaned and ready for use.

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4.2 Samples are extracted as soon as possible. When immediate extraction is not possible, samples are to be stored at 4°C for up to 14 days from the sampling date. When the Mass EPH and Mass TPH are required, samples must be extracted within seven days.

5.0 INTERFERENCES

5.1 Soxtherm glass thimbles, clamps, flasks and other glassware, must be carefully cleaned prior to each use in order to remove potential contaminants.

5.2 Solvents and reagents used in extraction must be quality control checked and proven free of interfering contaminants before acceptance for analytical use.

5.3 Phthalate esters will contaminate samples if plastic processing apparatus are used. Care should be taken to avoid any contact with plastic materials.

5.4 If necessary, Florisil and/or sulfur cleanup procedures may be employed. In such cases, proceed with SOP No. 50_3620B and/or SOP No. 50_3660B.

6.0 EQUIPMENT/APPARATUS AND MATERIALS

6.1 **Soxtherm automated extractor**: OI Analytical #981-012

6.2 **Glass thimbles:** OI Analytical / goes w/ OI # 8300

6.3 **TurboVap II** Concentration workstation (Zymark serial number: TV9915N8822)

6.3.1 Concentration tubes, 200 ml with 1.0 ml (part#: 45817)

6.4 **Boiling chips**: Solvent extracted, approximately 10/40 mesh Teflon.

6.5 **Vials**: Glass, 2-ml capacity, with Teflon lined screw caps or crimp tops.

6.6 **Glass Extraction Beaker**, 54x130mm. OI # 8300

6.7 **Heating mantle**: Rheostat controlled.

6.8 **Hamilton Calibrated Syringes**: 1-ml, 5-ml.

6.9 **Apparatus for determining percent moisture**:

6.9.1 Oven: Drying, VWR® Maintained at 105° C

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6.9.2 Desiccator, portable

6.9.3 Disposable aluminum weigh pans

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6.10 **Pasteur glass pipettes**: 1.0 ml, disposable.

6.11 **Beakers**: 150 ml.

6.12 **Calibrated Balance**: Top loading, OHAUS,® and Precision Standard TS-400

6.13 **Glass scintillation vials**: 20 ml, with Teflon lined screw caps.

6.14 **Spatula**: Stainless steel or Teflon.

6.15 1.0-ml glass bore **Hamilton Syringe**.

7.0 REAGENTS AND STANDARDS

7.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 Organic-free reagent water. All references to water in this method refer to organic-free reagent water, as defined in Chapter One, SW-846.

7.3 Sodium sulfate (granular, anhydrous), Na_2SO_4. Sodium sulfate is extracted and analyzed for contaminants before a lot is used. When necessary, the anhydrous sodium sulfate can be muffled in a furnace (4 hours) or solvent rinsed (i.e.: 4 hours with a soxhlet extraction apparatus) prior to use as a drying agent on sample extracts. Purify by heating at 400° C for 4 hours in a shallow tray, or by pre-cleaning the sodium sulfate with methylene chloride. If the sodium sulfate is pre-cleaned with methylene chloride, a method blank must be analyzed, demonstrating that there is no interference from the sodium sulfate. A record is kept in the Primary Reagent II Logbook (Attachment B).

7.4 Gauze Pads; Woundcare Solutions™, 2x2 ins. – 8 Ply, USP Type VII Gauze.

7.5 Extraction Solvents

7.5.1 Soil/sediment and aqueous sludge samples for PCB's and Pesticides shall be extracted using the following solvent system:

7.5.1.1 Acetone/Hexane ((1:1) (v/v)), $CH_3COCH_3/CH_3(CH_2)_4CH_2$, Pesticide quality or equivalent.

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7.5.2 Samples for Semi-Volatile Organics, EPH, and Total Petroleum Hydrocarbons shall be extracted using the following:

7.5.2.1 Methylene Chloride, CH_2Cl_2. Pesticide quality or equivalent.

7.4.2.2 Hexane, capillary GC/GC-MS solvent grade.

8.0 PROCEDURE

8.1 **Batching of samples:** All samples are set up as an analytical batch. The following must be true for samples to be considered a batch:

8.1.1 A batch consists of a group of 20 or less field samples of a similar matrix.

8.1.2 A preparation batch is defined as samples that are prepared together by the same person, or group of people, using the same equipment/glassware with the same method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in limited continuous sequential time periods, usually not to exceed 24 hours

8.2 Retrieve samples for analysis:

8.2.1 Review the ESS Organic Prep Back Log that was generated that morning.

8.2.2 Select 20 or fewer samples that require the 3541 extraction for a particular determinative method. If there are more than twenty samples select samples according to the following criteria

8.2.2.1 Sample which have hold times that are going to expire that day or the next should be analyzed first.

8.2.2.2 If hold times are not an issue then analyze samples according to their due date.

8.2.3 The samples are stored in the ESS walk-in cooler.

8.2.4 Log out all samples from the walk-in cooler. (Refer to the Sample Storage SOP 10_0002)

8.2.5 The batch of samples is transferred from the walk-in cooler, to the Organic Prep Lab. The samples are then placed in a ventilation hood in the organic prep lab.

8.2.6 All samples in the batch are logged into the Organic Prep Logbook (see Attachment A). The following information is documented:

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8.2.6.1 The ESS project numbers. This includes all projects that are being extracted in the batch.

8.2.6.2 Prep Date

8.2.6.3 The Blank ID which is also the batch ID.

Example: PXBE4112-BLK1

Where: P = the PCB determinative analysis
X = Soxtherm extraction
BE4112= the batch ID (assigned through ELEMENT)
BLK = Blank
1 = First blank for the analytical batch

Note: The prefix will change depending on the determinative method. PP = Pesticide, S = Semi volatile, T = TPH, E = EPH.

8.2.6.4 The analysis to be performed is checked off.

8.2.6.5 The analytical matrix is written as soil.

8.2.6.6 The extraction method is 3541

8.2.6.7 The ESS working standard IDs of the surrogates and spikes used are written into the blank spaces provided.

8.2.6.8 The ESS IDs of all samples to be extracted are documented. This includes QC samples.

8.2.6.9 The remainder is completed as the extraction is performed.

8.2.7 The batch is set up with the following QC standards:

8.2.7.1 **Method Blank**: An equivalent amount (to sample weight) of anhydrous Sodium Sulfate (or gauze pad for wipes) will be spiked with the appropriate surrogate, and placed in an extraction thimble, extracted, concentrated and analyzed like a sample. This is a quality control sample known to be free of any target analytes or contaminants. It is analyzed to verify that samples were not contaminated during extraction or analysis. The SVOA analyst will notify the organic preparation supervisor when contaminants are at levels that require re-extraction.

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8.2.7.2 **Blank Spike:** An equivalent amount (to sample weight) of anhydrous Sodium Sulfate (or gauze pad for wipes) will be spiked with the appropriate surrogate and matrix spike standard, and placed in an

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extraction thimble, extracted, concentrated and analyzed like a sample. This is a quality control sample that contains known concentrations of target analytes and is free from any sample matrix interference. It is analyzed to verify that under optimum conditions, without any sample matrix interference, the target analytes can be accurately recovered. If the recoveries of these target analytes are outside of established control limits then the entire batch must be re-extracted.

8.2.7.3 **Matrix Spike/Matrix Spike Duplicate:** One sample is randomly selected to be the sample, matrix spike and matrix spike duplicate. In some cases the client will specify which sample is to be used as the matrix spike and matrix spike duplicate sample. These are quality control samples, which contains known concentrations of target analytes in the sample matrix. They are analyzed to document the effect of the sample matrix on the recovery of the target analytes.

8.3 Sample Handling

8.3.1 Sediment/soil samples - Soil samples received for organic preparation typically have the consistency of coarse dry sand. In order to obtain a representative subsample for analysis, ESS Laboratory is guided by the subsampling techniques described in Attachment G of SOP 10.0001 (Sample Receiving), which calls for homogenization by stirring. Accordingly, the sample is thoroughly mixed, either in the original container, or if it should be necessary, by transferring to a larger clean glass container, and stirring with a clean glass rod. After mixing, weigh out an appropriate amount of sample (to the nearest 0.1 g) into a previously identified Soxhlet thimble. This is performed by discrete transfers from different parts of the sample container, avoiding any artifacts, such as rocks and twigs, and taking care to use all of the material transferred, in the analysis. In the event that the consistency differs from that described above, then Subsampling Attachment G, referred to above, must be consulted for further guidance. Record this weight in the logbook.

8.3.2 Solid materials – Samples reduced in size to fit in thimble. The sample must be submerged in solvent during extraction. (Includes concrete and wood)

8.3.3 Sludge: 5 g or less, dried with sodium sulfate.

8.4 Determination of percent solids: When sample results are to be calculated on a dry weight basis, a second portion of sample should be weighed at the same time as the portion used for analytical determination.

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WARNING - The drying oven should be contained in a hood or vented. Significant laboratory contamination may result from drying a heavily contaminated sample.

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8.4.1 Immediately after weighing the sample for extraction, weigh 10 g of the sample into a tared aluminum pan. Dry this aliquot at least 4 Hours at 105°C. Allow to cool in a desiccator before weighing. Calculate the % solids as follows:

$$\% \text{ Solids} = \frac{\text{dry weight X 100}}{\text{wet weight}}$$

8.4.2 Record the weights and percent solids in the Percent Solids Logbook (see Attachment C).

8.5 Sample Preparation for extraction: In the extraction thimble, containing a glass wool plug and enough Sodium sulfate to cover glass plug, weigh an appropriate amount of sample. If sample is very wet or is suspected to contain high levels of contaminants, smaller weight can be used. Keep in mind the affect on reporting limits. Some project specific procedures may require de-watering of samples prior to extraction. Refer to the project specific SOP. In the preparations log, document the weight to 0.1 g. Blend the indicated amount (see below) of the sample with anhydrous sodium sulfate. Keep adding sodium sulfate until a free flowing sandy dry mixture is achieved. The extraction thimble must drain freely for the duration of the extraction period.

Standard sample weights, in grams, are as follows:

Pesticides	20
PCBs (Soil)	20
PCBs (Concrete)	10
SVOA	15
LLPAH	15
TPH/DRO	20
EPH	25

8.6 Secure a metal clamp onto the top edge of the thimble. Place the thimble with clamp into an extraction beaker. ***Note: The metal clamp, the inside and outside surface of the thimble, and the inside surface of the extraction beaker must be kept clean.***

8.7 Add the surrogates listed in the determinative method to each sample and method blank. Add the surrogates and the matrix spike compounds appropriate for the project to the two additional aliquots of the sample selected for spiking and to the BS/BSD. If a drying agent is added to the field samples being extracted, it must also be added to the matrix spike aliquots, in order to assess the effect of the drying agent. Spiking requirements are as follows:

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Surrogates:

Class	Concentration, ppm	mls added	Final Volume (ml)
EPH	50.0	1.0	1.0
Pesticides	0.5	0.5	5.0
PCBs (soil/cement)	0.5	1.0	10.0

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SVOA	100	0.5	0.5
TPH/DRO	100	1.0	1.0
LLPAH	2.5	0.5	0.5

BS/BSD and MS/MSD:

Class	Concentration, ppm	mls added	Final Volume (ml)
EPH	50.0	1.0	1.0
Pesticides	0.5	0.5	5.0
PCBs (soil)	10.0	1.0	10.0
PCBs (cement)	10.0	1.0	10.0
SVOA	100.0	0.5	0.5
TPH/DRO	50.0	1.0	1.0
LLPAH	2.0	0.4	0.5

8.8 Immediately add 130 ml of the extraction solvent (Section 5.3) into the glass thimble. Slowly pour the solvent so that it drains through the thimble into the extraction beaker. 130 ml of solvent should be enough to cover the sample in the thimble. If the sample is not covered with solvent, the volume may need to be adjusted. Add two Teflon boiling chips to the solvent in the extraction beaker. Do not place the boiling chips in the thimble.

8.9 Place the extraction beaker containing the glass thimble with sample onto the automated Soxtherm extractor.

8.10 **Soxtherm Extractor Setup:**

8.10.1 Program the extraction method. The extraction method must be reviewed to ensure that it is accurate prior to every use. There are currently three programs in use; 1) Pesticides/PCBs; 2) SVOA, Oil and Grease by method 9071; 3) TPH/DRO/EPH. The following are the method programs:

Temperature Limit = 200 °C (Max temp of heater block) ***All methods***

Extraction Temperature = 1) 160 °C; 2) 140 °C; 3) 130 °C. Temperature at which heating blocks are controlled during sample processing. Should be 100-120°C above boiling point of extraction solvent.

Boiling Time = 1-2) 1 hour; 3) 40 minutes (Time in which thimble holding sample matrix is immersed in refluxing extraction solvent.)

Solvent reduction 1-2) A= 3 x 15; 3) A= 4 x 15 (Number of 15 ml solvent reduction to take place after Boiling time. Set to ensure the thimble is no longer immersed in extraction fluid.)

Extraction Time = 1-2) 50 min; 3) 40 min (time in which thimble holding the sample matrix is "rinsed" by the condensed solvent from boiling extraction solvent.)

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Solvent reduction 1) B = 1 x 15; 2) B = 0 x 15; 3) B = 2 x 15 (Number of 15 ml solvent reductions to take place after Extraction time is over. Chosen to achieve convenient final volume.)

Sol. Cooling = 1) 40 min; 2) 20 min; 3) 40 min (Timed period during which the heating block cools down while cooling water stays on. Used to avoid overheating concentrated extracts.)

Sol. Reduction interval = 4 sec (Length of interval between air pulses used to empty 15 ml condensed solvent into collection tank.) ***All methods***

Sol. Reduction pulse = 3 sec (length of air pulsed used to empty 15ml condensed solvent into collection tank) ***All methods***

8.10.2 The Soxtherm Automated Extractor sometimes reverts back to its default settings (Language, Date, and/or temperature settings change). Use the following steps to change functions on the Soxtherm controller:

8.10.2.1 Turn Power off

8.10.2.2 Turn power on and hold down the desired button (1 = Date and Time, 2 = Language E for English, 3 = Temperature control °C or °F)

8.10.2.3 When instructions appear, follow program instructions to edit function

8.10.2.4 Press enter to complete and save changes.

8.10.3 Prior to starting the Soxtherm the following procedures must be performed:

8.10.3.1 Turn on the gas flow.

8.10.3.2 Turn on the chillers (One chiller for every two units)

8.10.3.3 Turn on the control boxes.

8.10.3.4 On the control boxes turn on the flow. (The units are connected in series. All units must be turned on for gases and water to flow properly.)

8.10.3.5 Allow the gas and water flows to equilibrate for 5 minutes.

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8.10.4 To start the Soxtherm select the method utilizing the program in section 8.12.1. (Usually method 01.) Start each unit through the control box by selecting **R-1** and **Enter**. The extraction program is approximately 2.5 hours.

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8.10.5 Once the extracts have cooled, the extraction beakers with the thimbles are removed from the Soxtherm. The thimbles are removed from the extraction beakers. The fluid remaining in the extraction beakers is the extract. The final volume will be between 20 ml and 30 ml. Next, the extracts will need to be concentrated to final volumes.

8.11 TurboVap Extract Concentration Technique.

8.11.1 The TurboVap technique uses the Zymark TurboVap II concentration workstation. This system uses a warm water bath and a continuous flow of nitrogen to concentrate samples. Turn on the TurboVap using the button on the lower left-hand side. Fill water bath and set bath temperature to 40-45°C. The TurboVap can be programmed to stop after a certain time, manually, or using an optical sensor. When the sensor is used, the TurboVap will stop ("beep") when the sample extract reaches 0.75 ml. For dirty samples, the "time" option should be used due to false sensor readings. Adjust the gas pressure using the knob on the left hand side (behind the power button). Pull knob out and turn clockwise to increase pressure and counterclockwise to decrease pressure. The pressure should be set at 10-15 psi. For samples that are 200ml or greater, start at a lower pressure then increase when sample has evaporated below 200ml mark on concentrator tube. This will eliminate the risk of sample loss due to splashing.

8.11.2 In most cases, if enough sodium sulfate was used to dry the sample, the extract will be anhydrous. Continue with section 8.13.6. When the extracts appear to contain water the extract will have to be dried. To dry the extract, place a glass funnel into the top of the concentrator tube. Use a small piece of glass wool to plug the funnel. Pour approximately 100g of dry sodium sulfate into the funnel.

8.11.3 Quantitatively pour the sample into the funnel. The sample should filter through the sodium sulfate and glass wool. All water in the sample should be absorbed by the sodium sulfate. No water should pass into the tube. Re-filter if water is present in the tube.

8.11.4 To ensure that the entire sample has been transferred into the concentration tube, rinse the flask containing the original extract with approximately 5 ml of the solvent used for extraction. All rinsate should be collected in the concentration tube. The concentrator tube should only be filled to 200 ml. If more extract remains, evaporate a portion of the extract in the tube then decant the rest of the extract. Transfer the ESS Lab ID to the concentrator tube.

8.11.5 Remove the funnel containing sodium sulfate. The sodium sulfate and glass wool in the funnel is disposed of in the laboratory solid waste container.

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8.11.6 Place concentrator tubes with extract into the TurboVap. Adjust bath temperature and gas flow and start by pressing the appropriate cell number (1-6).

8.11.7 Concentration procedure (Analysis specific)

8.11.7.1 When samples are being analyzed **for EPH**, a solvent exchange is required. The TurboVap will "beep" when extract has reached 0.75 ml or when the preset time has expired. When the original extract has been evaporated to 0.75 ml, add approximately 30 ml of hexane and evaporate to 0.75 ml. Using a syringe, bring the final volume up to 1.0 ml using Hexane. The sample is now ready for the fractionation procedure. Refer to the EPH SOP.

8.11.7.2 When samples are being analyzed for Pesticides and PCBs, the final extract volume will be 10 ml. The Hexane/Acetone extract is concentrated to just under 10 ml. Using a glass pipette, the extract is transferred to a 15 ml vial. The TurboVap cell is then rinsed with Hexane. The rinsate is also transferred to the 15 ml vial. The solvent is then quantitatively brought up to a final volume of 10 ml. A graduated cylinder is used to determine the final volume.

8.11.7.3 When SVOA, TPH, and DRO are being concentrated, the final extract volume should be 1.0 ml. After the TurboVap has finished concentrating to 0.75 ml, bring the extract to 1.0 ml with the solvent used for extraction using a calibrated syringe and quantitatively transfer to a 2.0 ml target vial. The ESS Lab ID is transcribed to the target vial.

8.11.8 The extract is now ready to be analyzed for the target analytes using the appropriate determinative technique(s). All extracts must be analyzed within 40 days.

9.0 CALCULATIONS

9.1 Not applicable: See determinative method.

10.0 QUALITY CONTROL

10.1 Before processing any samples, the analyst should demonstrate that all parts of the equipment in contact with the sample and reagents are interference-free. This is accomplished through the analysis of a solid matrix method blank (see Section 8.2.7.1). Each time samples are extracted, and when there is a change in reagents, a method blank (use same amount as sample weight) needs to be extracted and analyzed for the compounds of interest. Use same amount as sample weight. The method blank should be carried through all stages of the sample preparation and measurement.

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10.2 Standard quality assurance practices should be used with this method. Field duplicates should be collected to validate the precision of the sampling procedures. A matrix spike/matrix spike duplicate, or matrix spike and duplicate sample analysis, and a laboratory control sample should be prepared and analyzed with each batch of samples prepared by this procedure, unless the determinative method provides other guidance.

10.3 Surrogate and spike standards should be added to samples according to the appropriate determinative method.

10.4 A batch is a group of less than or equal to 20 samples prepared on a given day.

11.0 DATA VALIDATION

11.1 Refer to the determinative methods for performance data.

11.2 Any unusual observations are noted in the prep log.

11.3 All logbooks are routinely reviewed by the laboratory supervisor.

11.4 All non-compliances require a corrective action report. This report must be included with the raw data. Refer to the quality control of the determinative method.

12.0 REFERENCES

12.1 SW846 Method 3541, (09/94) Automated Soxhlet Extraction, third edition. Update III, Volume 1B, chap. 4, sec. 4.2.1

12.2 NELAC/NELAP Chapter 5, June 2003

12.3 DoD QSM, Final Version 3, January 2006

12.4 ESS Laboratory Policies and Procedure

13.0 POLLUTION PREVENTION and WASTE MANAGEMENT

13.1 ESS Laboratory's policies on pollution prevention and waste management are covered in SOP 90_0002, Hazardous Waste Contingency and Emergency Response Plan. All employees are trained in the requirements of the SOP.

14.0 DEFINITIONS

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14.1 Internal Standard - A pure analyte(s) added to a sample, extract, or standard solution in known amount(s) and used to measure the relative responses of other method analytes and surrogates that are components of the same sample or solution. The internal standard must be an analyte that is not a sample component.

14.2 Surrogate Analyte - A pure analyte(s), which is extremely unlikely to be found in any

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sample, and which is added to a sample aliquot in known amount(s) before extraction or other processing and is measured with the same procedures used to measure other sample components. The purpose of the SA is to monitor method performance with each sample.

14.3 Laboratory Duplicate – A second aliquot of a sample taken in the laboratory and analyzed separately with identical procedures as the sample. Analyses of the lab duplicate and sample indicates precision associated with laboratory procedures, but not with sample collection, preservation, or storage procedures.

14.4 Field Duplicates - Two separate samples collected at the same time and place under identical circumstances and treated exactly the same throughout field and laboratory procedures. Analyses of the sample and its field duplicate give a measure of the precision associated with sample collection, preservation and storage, as well as with laboratory procedures.

14.5 Method Blank - An aliquot of reagent water or other blank matrix that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The Method Blank is used to determine if method analytes or other interferences are present in the laboratory environment, reagents, or the apparatus.

14.6 Field Blank (FB) - An aliquot of reagent water or other blank matrix that is placed in a sample container in the laboratory and treated as a sample in all respects, including shipment to the sampling site, exposure to sampling site conditions and equipment, storage, preservation, and all analytical procedures. The purpose of the FB is to determine if method analytes or other interferences are present in the field environment.

14.7 Blank Spike (BS) - An aliquot of reagent water or other blank matrix to which known quantities of the method analytes are added in the laboratory. The BS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.

14.8 Matrix Spike (MS) - An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The MS is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS corrected for background concentrations.

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14.9 Stock Standard Solution - A concentrated solution containing one or more method analytes prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source.

14.10 Calibration Standard (CAL) - A solution prepared from the primary dilution standard

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solution or stock standard solutions and the internal standards and surrogate analytes. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

14.11 Quality Control Sample - A solution of method analytes of known concentrations which is used to fortify an aliquot of BS or sample matrix. The quality control sample (known as second source standard) is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check laboratory performance with externally prepared test materials.

15.0 METHOD PERFORMANCE

15.1 See determinative methods for method performance information.

16.0 PERSONNEL QUALIFICATIONS

16.1 BA/BS Chemistry, Natural & Applied Science or equivalent experience.

16.2 All analysts, before performing any analysis, participate in the ESS Laboratory training program (SOP80_0016). The training process consists of reading the Standard Operating Procedure, gaining instruction on the procedure from an experienced analyst, and performing the initial demonstration of capability.

17.0 TROUBLESHOOTING

17.1 See Determinative method.

18.0 DATA MANAGEMENT AND RECORDS

18.1 **Data Management** - ESS Laboratory's utilizes the Promium Element LIMS system as part of its Data Management system. Client sample information is entered into ELEMENT LIMS and analyses are assigned to each sample. The LIMS allows EPA hold times, minimum batch QC requirements, and QC criteria to be assigned to each analysis. Standards can be entered and assigned to QC samples through the LIMS. Once analysis has been performed, data is imported using DataTool avoiding manual errors. In conjunction with Crystal Reports, the ELEMENT system allows for a wide variety of reporting formats.

18.2 **Records** – The specific retention periods required in the NELAC Standards, EPA-CFR and state and local statutes are followed or exceeded. At a minimum, data records are retained for five years from last use (10 years for drinking water). If there is a question about whether a record should be retained or disposed because no specific requirement could be found, the record is retained until such time as a retention period is specified. Records are stored in specified-labeled locations and are easily retrievable. All raw data associated with testing is also retained including; computer printouts, chromatograms, review forms, and logbooks.

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19.0 ATTACHMENTS

19.1 Attachment A – Organic Prep logbook

19.2 Attachment B – Primary Reagent II Logbook

19.3 Attachment C – Percent Solids logbook

19.4 Addendum 1 – Modification describing the extraction for MA-MCP Pesticides.

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Attachment A

ESS Organic Preparation Logbook

Project #:____________
Prep Date:____________
Batch ID:____________
Extraction Method:____________

Surrogate ID#
A____________
B____________
C____________

Matrix Spike ID#
D____________
E____________
F____________

Analytical Matrix:____________
Extraction Time:
Start:________
Finish:________

Split Extraction* ☐
* Half of the final extract volume (0.5ml) is exchanged into 5ml 5ml hexane and transferred as Vol 1. The other half (0.5ml CH_2Cl_2) is transferred as Volume 2.

ESS ID	Vol(ml)/ Wt.(g)	Surrogate (ul or ml)	Matrix Spike (ul or ml)	Final Extract Vol (ml) Hex/CH_2Cl_2	Transfer Vol #1 (ml) Hex/CH_2Cl_2	Transfer Vol #2 (ml) Hex/CH_2Cl_2	Transfer Date	Bath Temp (C)	pH	Discard bottle**	Comments	1st Rvw Init.	Witness Init.	2nd Rvw Init.

Analysis Performed
- PCB ☐
- B/N SVOA ☐
- SVOA ☐
- LL PAH ☐
- PEST ☐
- TPH/GC ☐
- BIS-2 ☐
- PAH ☐

Acid Washed: Y / N	Cu Cleaned: Y / N	Florisiled: Y / N	Silica Column/Carbon prep: Y / N
H_2SO_4 ID#	Cu ID#	Lot#	Lot #

CH_2Cl_2 lot #____________ NaOH ID#____________

Prepared By:____________ Glasswool:____________ Method #(s):____________ Hexane lot#____________ Na_2SO_4 ID#____________

**Check off column if entire sample used and bottle discarded. Acetone lot#____________

Control #50.0001-0701A **BATCH ID/Test:**____________ **BATCH ID/Test:**____________ Page________

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Primary Reagent Log II

* for Primary Reagents treated in Muffle or Drying Oven

Date	Lab ID #	Reagent Name	1° Lab ID	Preparation			Exp. Date	1st Rvw Init.
				Oven Used	Temp. (°C)	Duration		

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Reviewed by:_____ Control #50.0018-0702A Page_____

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ATTACHMENT C

ESS LABORATORY
Percent Solids Logbook

Date/ Time	Lab ID	Pan WT. (g)	Wet WT. (g)	Dry WT. (g)	Percent Solid	Wet wt. Init.	Dry wt./1st Rvw Init.	2nd Rvw Init.

Criteria: Dup RPD ≤ 20%

Control #50.0006-0701A

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Addendum I

3541 SOP Modification

SOP Modified: Automated Soxhlet Extraction
Technique: ESS Laboratory SOP 50_3541 Rev. 3

Modification Objective: This addendum addresses any modification to Standard Operating Procedure 50_3541 needed to extract samples for MA-MCP Pesticides.

Specific SOP Modifications: Method SW846 3541 includes the following changes:

- Section 8.7: Add 1.0 ml (0.5 ml for MA-MCP Pesticides) of the surrogate standard spiking solution onto the sample.

- Section 8.11.7.3: When samples are being analyzed for Pesticides and PCBs, the final extract volume will be 10 ml (5 ml for MA-MCP Pesticides). The Hexane/Acetone extract is concentrated to just under 10 ml (5 ml for MA-MCP Pesticides). Using a glass pipette, the extract is transferred to a 15 ml vial. The TurboVap cell is then rinsed with Hexane. The rinsate is also transferred to the 15 ml vial. The solvent is then quantitively brought up to a final volume of 10 ml (5 ml for MA-MCP Pesticides). A graduated cylinder is used to determine the final volume.

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ESS Laboratory
Division of Thielsch Engineering
Cranston, RI

SOP NO. 50_3546
MICROWAVE EXTRACTION
(SW-846 Method 3546)

REVIEWED BY:

Operations Manager	Date 8/14/07
QA Officer	Date 8/14/07
Laboratory Director	Date

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MICROWAVE EXTRACTION
(SW-846 Method 3546)

1.0 SCOPE AND APPLICATION

1.1 Method 3546 is a procedure for extracting water insoluble or slightly water soluble organic compounds from soils, clays, sediments, sludges, and solid wastes. The method was developed and validated on commercially-available solvent extraction systems. The procedure uses microwave energy to produce elevated temperature and pressure conditions (i.e., 100 - 115°C and 50 - 175 psi) in a closed vessel containing the sample and organic solvent(s) to achieve analyte recoveries equivalent to those from Soxhlet extraction (SW846 Method 3540), using less solvent and taking significantly less time than the Soxhlet procedure.

1.2 This method is applicable to the extraction of semivolatile organic compounds, organophosphorus pesticides, organochlorine pesticides, chlorinated herbicides, phenoxyacid herbicides, substituted phenols, PCBs, and PCDDs/PCDFs, which may then be analyzed by a variety of chromatographic procedures. The method may also be applicable for the extraction of additional target analytes, provided that the analyst demonstrates adequate performance for the intended application, per SW846 Method 3500 and SW846 Chapter Two.

1.3 This method has been validated for solid matrices containing 50 to 10,000 µg/kg of semivolatile organic compounds, 250 to 2,500 µg/kg of organophosphorus pesticides, 10 to 5,000 µg/kg of organochlorine pesticides and chlorinated herbicides, 50 to 2,500 µg/kg of substituted phenols, 100 to 5,000 µg/kg of phenoxyacid herbicides, 1 to 5,000 µg/kg of PCBs, and 10 to 6000 ng/kg of PCDDs/PCDFs. The method may be applicable to samples containing these analytes at higher concentrations and may be employed after adequate performance has been demonstrated for the concentrations of interest.

1.4 This method only is applicable to solid samples with small particle sizes. If practical, soil/sediment samples may be air-dried and ground to a fine powder prior to extraction. Alternatively, if worker safety or the loss of analytes during drying is a concern, soil/sediment samples may be mixed with anhydrous sodium sulfate or pelletized diatomaceous earth. The total mass of material to be prepared depends on the specifications of the determinative method and the sensitivity required for the analysis, but 2 - 20 g of material are usually necessary and can be accommodated by this extraction procedure.

1.5 This method has been validated using a solvent mixture of hexane and acetone (1:1) from matrices, such as soil, glass-fibers and sand. Other solvent systems may be employed, provided that adequate performance can be demonstrated for the analytes of interest (refer to SOP 80.0024 entitled "Process Validation").

1.6 Prior to employing this method, analysts are advised to consult the base method and SW846 Chapter 2 for each type of procedure that may be employed in the overall analysis (e.g., SW846 Methods 3500, 3600, 5000, and 8000 may be found in the network directory

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Q:\Quality\Methods\SW846), for additional information on quality control procedures, development of QC acceptance criteria, calculations, and general guidance on the intended flexibility in the choice of methods, apparatus, materials, reagents, and supplies, and on the responsibilities of the analyst for demonstrating that the techniques employed are appropriate for the analytes of interest, in the matrix of interest, and at the levels of concern.

1.7 This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 METHOD SUMMARY

2.1 Samples are prepared for extraction by mixing with a drying agent, and loaded into the extraction vessel.

2.2 The appropriate solvent system is added to the vessel which is then sealed.

2.3 The extraction vessel containing the sample and solvent system is heated to the extraction temperature and microwaved for the specified period of time.

2.4 The mixture is allowed to cool. The vessel is opened and the contents are filtered. The solid material is rinsed and the various solvent fractions are combined.

2.5 The extract may be concentrated, if necessary, and, as needed, exchanged into a solvent compatible with the cleanup or determinative procedure being employed.

3.0 HEALTH AND SAFETY

3.1 Each employee has been trained and has acknowledged being trained in the safe use and handling of chemicals. This training has been performed according to the ESS Training SOP 80_0016 and by the Chemical Hygiene Plan, SOP No. 90_0001, in conjunction with the Safety orientation.

3.2 All sample and material handling should be done in a hood while using proper protective equipment to minimize exposure to liquid or vapor. Minimum personnel protective equipment includes the use of laboratory safety glasses, a lab coat or apron, and protective gloves.

3.3 The MSDS for the concentrated chemicals used in the laboratory are kept on file in a central location that is available for all employees to review.

4.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

4.1 Sample containers will be borosilicate glass with Teflon lined screw caps, typically 250-ml wide mouth amber borosilicate bottles. They will be purchased pre-cleaned and ready for use.

4.2 Samples are extracted as soon as possible. When immediate extraction is not possible, samples are to be stored at 4°C for up to 14 days from the sampling date.

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5.0 INTERFERENCES

5.1 Microwave vessels and glassware, must be carefully cleaned prior to each use in order to remove potential contaminants.

5.2 Solvents and reagents used in extraction must be quality control checked and proven free of interfering contaminants before acceptance for analytical use.

5.3 Phthalate esters will contaminate samples if plastic processing apparatus are used. Care should be taken to avoid any contact with plastic materials.

5.4 If necessary, Florisil and/or sulfur cleanup procedures may be employed. In such cases, proceed in accordance with SOP No. 50.3620B and/or SOP No. 50.3660B.

6.0 EQUIPMENT/APPARATUS

6.1 **Data system:**

6.1.1 Computers: The OP laboratory has an AST computer with a Windows 95 operating system. All computer systems are networked to a Windows 2000 server, which is the destination of all files. A differential back-up is performed nightly and a full back is performed each weekend using Veritas Backup Exec to tapes. As the systems acquires and stores data onto the server, the server becomes full. The data is downloaded and archived onto CDs.

6.2 **Microwave solvent extraction apparatus**

6.2.1 The temperature performance requirements necessitate that the microwave extraction system be capable of sensing the temperature to within ± 2.5°C and automatically adjusting the microwave field output power within 2 seconds of sensing. Temperature sensors should be accurate to ± 2°C. Temperature feedback control provides the primary performance mechanism for the method. The instrument used by ESS Laboratory is a CEM Microwave Extractor, which meets these requirements.

6.2.2 Microwave extraction vessels capable of accommodating 1g to 20 g samples, transparent to microwave energy, relatively inert to reagents and sample components and capable of withstanding the temperature and pressure requirements (minimum conditions of 200°C and 200 psi), necessary to perform this procedure, are supplied by CEM with the extractor. Manufacturer's instructions regarding cleaning, handling, and sealing the vessels are followed.

6.3 **TurboVap II** Concentration workstation.

6.3.1 Concentration tubes, 200 ml with 1.0 ml.

6.4 **Boiling chips**: Solvent extracted, approximately 10/40 mesh Teflon.

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6.6 **Glass Extraction Beaker**, 54x130mm. OI # 8300.

6.7 **Deleted**

6.8 **Hamilton Calibrated Syringes**: 1 ml, 5 ml.

6.9 **Apparatus for determining percent moisture**:

6.9.1 Oven: Drying, VWR® Maintained at 105° C.

6.9.2 Desiccator, portable.

6.9.3 Disposable aluminum weigh pans.

6.10 **Pasteur glass pipettes**: 1.0 ml, disposable.

6.11 **Beakers**: 150 ml.

6.12 **Calibrated Balance**: Top loading, OHAUS,® and Precision Standard TS-400.

6.13 **Glass scintillation vials**: 20 ml, with Teflon lined screw caps.

6.14 **Spatula**: Stainless steel or Teflon.

6.15 1.0-ml glass bore **Hamilton Syringe**.

7.0 REAGENTS AND STANDARDS

7.1 Reagent grade chemicals are used in all tests. Unless otherwise indicated, these reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 Organic-free reagent water. All references to water in this method refer to organic-free reagent water as defined in SW846 Chapter One, which is prepared as described in SOP 110.0003.

7.3 Drying agents

7.3.1 Sodium sulfate (granular, anhydrous), Na2SO4. Sodium sulfate is extracted and analyzed for contaminants before a lot is used. When necessary, the anhydrous sodium sulfate can be muffled in a furnace (4 hours) or solvent rinsed (i.e.: 4 hours with a Soxhlet extraction apparatus) prior to use as a drying agent for1 sample extracts; purify by heating at 400° C for 4 hours in a shallow tray, or by pre-cleaning the sodium sulfate with methylene chloride. If the sodium sulfate is pre-cleaned with methylene chloride, a method blank must be analyzed,

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demonstrating that there is no interference from the sodium sulfate. A record is kept in the Primary Reagent II Logbook (Attachment B).

7.4 Extraction solvents

7.4.1 This method has been validated using a 1:1 mixture of hexane and acetone from matrices such as soil, glass-fibers, and sand. Other solvent systems may have applicability in microwave extraction, provided that at least one component absorbs microwave energy. The choice of extraction solvent will depend on the analytes of interest and no single solvent is universally applicable to all analyte groups. Whatever solvent system is employed, including those specifically listed in this method, the analyst must demonstrate adequate performance for the analytes of interest, at the levels of interest. At a minimum, such a demonstration will encompass the initial demonstration of proficiency described in Sec. 8.2 of SW846 Method 3500, using a clean reference matrix. Hexane is a water-immiscible solvent and acetone is a water-miscible solvent. The purpose of the water-miscible solvent is to facilitate the extraction of wet solids by allowing the mixed solvent to penetrate the layer of water of the surface of the solid particles. The water-immiscible solvent extracts organics compounds with similar polarities. The polarity of acetone may also help extract polar analytes in mixed solvent systems.

7.4.2 Methylene Chloride, CH_2Cl_2, has also been validated for use in certain determinative methods.

7.4.3 Hexane.

7.4.4 All solvents should be pesticide quality or equivalent. Solvents may be degassed prior to use.

8.0 CALIBRATION AND STANDARDIZATION

There are no calibration or standardization steps associated with this sample extraction procedure other than establishing the extraction conditions in Section 9.11.

9.0 PROCEDURE

9.1 **Batching of samples:** All samples are set up as an analytical batch. The following must be true for samples to be considered a batch:

9.1.1 A batch consists of a group of 20 or less field samples of a similar matrix.

9.1.2 A preparation batch is defined as samples that are prepared together by the same person, or group of people, using the same equipment/glassware with the same method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in limited continuous sequential time periods, usually not to exceed one 24 hour period.

9.2 **Retrieve samples for analysis:**

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9.2.1 Review the ESS Organic Prep Back Log which is generated daily.

9.2.2 Select 20 or fewer samples that require the 3546 extraction for a particular determinative method. If there are more than twenty samples select samples according to the following criteria:

9.2.2.1 Sample having hold times that are about to expire should be analyzed first.

9.2.2.2 If hold times are not an issue then samples are to be analyzed according to their due date.

9.2.3 The samples are stored in the ESS walk-in cooler.

9.2.4 Log out all samples from the walk-in cooler. (Refer to the Sample Storage SOP 10.0002)

9.2.5 The batch of samples is transferred from the walk-in cooler, to the Organic Prep Lab. The samples are then placed in a ventilation hood in the organic prep lab.

9.2.6 All samples in the batch are logged into the Organic Prep logbook (see Attachment A). The following information is documented:

9.2.6.1 The ESS project numbers. This includes all projects that are being extracted in the batch.

9.2.6.2 Prep Date

9.2.6.3 The Blank ID which is also the batch ID.

Example: PMBE4112-BLK1

Where: P = the PCB determinative analysis
M = Microwave extraction
BE4112= the batch ID (assigned through ELEMENT)
BLK = Blank
1 = First blank for the analytical batch

Note: The prefix will change depending on the determinative method. PP = Pesticide, S = Semi volatile, T = TPH, E = EPH.

9.2.6.4 The analysis to be performed is checked off.

9.2.6.5 The analytical matrix is written as soil.

9.2.6.6 The extraction method is written in as 3546.

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9.2.6.7 The ESS working standard IDs of the surrogates and spikes used are written into the blank spaces provided.

9.2.6.8 The ESS IDs of all samples to be extracted are documented. This includes QC samples.

9.2.6.9 The remainder is completed as the extraction is performed.

9.2.7 The batch is set up with the following QC standards:

9.2.7.1 **Method Blank**: Sodium Sulfate will be spiked with the appropriate surrogate, and placed in an extraction vessel, extracted, concentrated and analyzed like a sample. This is a quality control sample known to be free of any target analytes or contaminants. It is analyzed to verify that samples were not contaminated during extraction or analysis. The SVOA analyst will notify the organic preparation supervisor when contaminants are at levels that require re-extraction.

9.2.7.2 **Blank Spike:** Sodium Sulfate will be spiked with the appropriate surrogate and matrix spike standard, and placed in an extraction vessel, extracted, concentrated and analyzed like a sample. This is a quality control sample that contains known concentrations of target analytes and is free from any sample matrix interference. It is analyzed to verify that under optimum conditions, without any sample matrix interference, the target analytes can be accurately recovered. If the recoveries of these target analytes are outside of established control limits then the entire batch must be re-extracted.

9.2.7.3 **Matrix Spike/Matrix Spike Duplicate:** One sample is randomly selected to be the sample, matrix spike and matrix spike duplicate. In some cases the client will specify which sample is to be used as the matrix spike and matrix spike duplicate sample. These are quality control samples, which contains known concentrations of target analytes in the sample matrix. They are analyzed to document the effect of the sample matrix on the recovery of the target analytes.

9.3 Sample preparation

The sample preparation steps vary with the type of sample to be extracted, as described below. samples are mixed with a drying agent such as sodium sulfate, prior to extraction.

9.3.1 Sediment/soil samples

Soil samples received for organic preparation typically have the consistency of coarse dry sand. In order to obtain a representative subsample for analysis, ESS Laboratory is guided by the subsampling techniques described in Attachment G of SOP 10.0001 (Sample Receiving), which calls for homogenization by stirring. Accordingly, the sample is thoroughly mixed, either in the original container, or if it should be necessary, by transferring to a larger clean glass container, and stirring with a clean glass rod. After mixing, weigh out an appropriate amount (see § 9.5) of sample (to the

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nearest 0.1 g) into a previously identified extraction vessel. This is performed by discrete transfers from different parts of the sample container, avoiding any artifacts, such as rocks and twigs, and taking care to use all of the material transferred, in the analysis. In the event that the consistency differs from that described above, then Subsampling Attachment G, referred to above, must be consulted for further guidance. Record this weight in the logbook.

NOTE: Dry, finely-ground soil/sediment allows the best extraction efficiency for nonvolatile, nonpolar organics, e.g., 4,4'-DDT, PCBs, etc.

NOTE: Mixing should always be performed in a hood, to avoid contamination of the laboratory.

9.3.2 Waste samples

Multiphase waste samples must be prepared by the phase separation method in SW-846 Chapter Two before extraction. The microwave extraction procedure is for solids only.

9.3.3 Dry sediment/soil and dry waste samples amenable to grinding
Grind or otherwise reduce the particle size of the waste so that it either passes through a 1-mm sieve or can be extruded through a 1-mm hole. Disassemble grinder between samples, according to manufacturer's instructions, and decontaminate with soap and water, followed by acetone and hexane rinses. The notes in Sec. 11.1 also apply to the grinding process.

9.3.4 Gummy, fibrous, or oily materials not amenable to grinding
Cut, shred, or otherwise reduce in size these samples to allow mixing and maximum exposure of the sample surfaces for the extraction. The analyst may add anhydrous sodium sulfate to the sample to make it more amenable to grinding.

9.4 Determination of percent dry weight

When sample results are to be calculated on a dry weight basis, a second portion of sample should be weighed at the same time as the portion used for analytical determination.

WARNING: The drying oven should be contained in a hood or vented. Significant laboratory contamination may result from drying a heavily contaminated sample.

9.4.1 Immediately after weighing the sample for extraction, weigh 5-10 g of the sample into a tared crucible. Dry this aliquot overnight at 105°C. Allow to cool in a desiccator before weighing.

9.4.2 Calculate the % dry weight as follows:

$$\% \text{ dry weight} = \frac{\text{g dry sample}}{\text{g of sample}} \times 100$$

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9.4.3 Record the weights and percent solids in the Percent Solids Logbook (see Attachment C).

9.5 Transfer the sample (Sect.9.3) to an extraction vessel. The weight of a specific sample that a vessel will contain depends on the bulk density of the sample and the amount of drying agent (if any) that was added to the sample in order to make it suitable for extraction. Analysts should ensure that the sample aliquot extracted is large enough to provide the necessary sensitivity.

Standard sample weights, in grams, are as follows:

Pesticides	20
PCBs (Soil)	20
PCBs (Concrete)	10
SVOA	15
LLPAH	15
TPH/DRO	20
EPH	25

9.6 Prepare a method blank using an aliquot of a clean solid matrix such as sodium sulfate, of the approximate weight of the samples. If a drying agent is added to the field samples being extracted, it must also be added to the method blank, in order to assess the possible contribution of the drying agent to the blank results.

9.7 Add the surrogates listed in the determinative method to each sample and method blank. Add the surrogates and the matrix spike compounds appropriate for the project to the two additional aliquots of the sample selected for spiking and to the BS/BSD. If a drying agent is added to the field samples being extracted, it must also be added to the matrix spike aliquots, in order to assess the effect of the drying agent. Spiking requirements are as follows:

Surrogates:

Class	Concentration, ppm	mls added	Final Volume (ml)
EPH	50.0	1.0	1.0
Pesticides	0.5	0.5	5.0
PCBs (soil/cement)	0.5	1.0	10.0
SVOA	100	0.5	0.5
TPH/DRO	100	1.0	1.0
LLPAH	2.5	0.5	0.5

BS/BSD and MS/MSD:

Class	Concentration, ppm	mls added	Final Volume (ml)
EPH	50.0	1.0	1.0
Pesticides	0.5	0.5	5.0
PCBs (soil)	10.0	1.0	10.0
PCBs (cement)	10.0	1.0	10.0
SVOA	100.0	0.5	0.5
TPH/DRO	50.0	1.0	1.0
LLPAH	2.0	0.4	0.5

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9.8 Add approximately 25 - 30 ml of the appropriate solvent system to the vessel and seal the vessel as instructed by the manufacturer.

Solvents:

Pesticides/PCBs – Hexane/Acetone (1:1)
EPH/TPH/DRO/SVOA/LLPAH – CH_2Cl_2

9.9 Place the extraction vessel into the microwave apparatus and proceed with setup as instructed by the instrument manufacturer. Vessels are to be evenly arranged in the apparatus, filling the inner ring first.

9.10 Extraction conditions (programmed):

Ramp up to 115°C over a period of about 10 minutes.
Temperature: 115°C.
Time at Temperature: 10 minutes.
Cooling: To room temperature over a period of about 15 minutes.
Filtering/Rinsing: With same solvent system.

9.10.1 The time/temperature conditions may be optimized, as needed, according to the manufacturer's instructions. In general, the pressure is not a critical parameter, since it is a result of the solvent system vapor pressure at the elevated temperature.

9.10.2 Once established, the same procedure should be used for all samples extracted for the same type of analysis. Any changes must be validated and documented in this SOP.

9.10.3 The amount of power used for extraction must be adjusted according to the number of samples extracted. Running the system at full power with just a few samples will cause the samples to overheat and loose most of the solvent. Four methods were created for different sample volumes.

Method	Max Power	%Power	Time	# of samples
3546	1600W	100	10	20+
3546 Med	1600W	75	10	10-20
3546 Small	800W	100	10	8-10
xSmall	1600W	25	10	<8

9.11 Begin the extraction according to the instructions provided by the manufacturer.

9.12 Allow the extracts to cool to room temperature once the extractions are complete. After cooling, open the vessels and proceed with filtering and rinsing, combining all the filtrates.

9.13 The extract is now ready for concentration, cleanup, or analysis, depending on the presence of interferants and the determinative method to be employed. Refer to SW846 Method 3600 for guidance on selecting appropriate cleanup methods. Excess water present in extracts may be removed by filtering the extract through a bed of anhydrous sodium sulfate. Certain cleanup and/or determinative methods may require a solvent exchange prior to cleanup and/or

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analysis.

9.14 **TurboVap Extract Concentration Technique.**

9.14.1 The TurboVap technique uses the Zymark TurboVap II concentration workstation. This system uses a warm water bath and a continuous flow of nitrogen to concentrate samples. Turn on the TurboVap using the button on the lower left-hand side. Fill water bath and set bath temperature to 40-45°C. The TurboVap can be programmed to stop after a certain time, manually, or using an optical sensor. When the sensor is used, the TurboVap will stop ("beep") when the sample extract reaches 0.75 ml. For dirty samples, the "time" option should be used due to false sensor readings. Adjust the gas pressure using the knob on the left hand side (behind the power button). Pull knob out and turn clockwise to increase pressure and counterclockwise to decrease pressure. The pressure should be set at 10-15 psi. For samples that are 200 ml or greater, start at a lower pressure then increase when sample has evaporated below 200 ml mark on concentrator tube. This will eliminate the risk of sample loss due to splashing.

9.14.2 In most cases, if enough sodium sulfate was used to dry the sample, the extract will be anhydrous. Continue with Section 9.15.6. When the extracts appear to contain water the extract will have to be dried. To dry the extract, place a glass funnel into the top of the concentrator tube. Use a small piece of glass wool to plug the funnel. Pour approximately 100 g of dry sodium sulfate into the funnel.

9.14.3 Quantitatively pour the sample into the funnel. The sample should filter through the sodium sulfate and glass wool. All water in the sample should be absorbed by the sodium sulfate. No water should pass into the tube. Re-filter if water is present in the tube.

9.14.4 To ensure that the entire sample has been transferred into the concentration tube, rinse the flask containing the original extract with approximately 5 ml of the solvent used for extraction. All rinsate should be collected in the concentration tube. The concentrator tube should only be filled to 200 ml. If more extract remains, evaporate a portion of the extract in the tube then decant the rest of the extract. Transfer the ESS Lab ID to the concentrator tube.

9.14.5 Remove the funnel containing sodium sulfate. The sodium sulfate and glass wool in the funnel is disposed of in the laboratory solid waste container.

9.14.6 Place concentrator tubes with extract into the TurboVap. Adjust bath temperature and gas flow and start by pressing the appropriate cell number (1-6).

9.14.7 Concentration procedure (Analysis specific)

9.14.7.1 When samples are being analyzed **for EPH**, a solvent exchange is required. The TurboVap will "beep" when extract has reached 0.75 ml or when the preset time has expired. When the original extract has been evaporated to 0.75 ml, add approximately 30 ml of hexane and evaporate to 0.75 ml. Using a calibrated Hamilton syringe, bring the final volume up to 1.0 ml using Hexane. The sample is now ready for the fractionation procedure. Refer to SOP 60.8100 Mod.

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9.14.7.2 When samples are being analyzed for Pesticides and PCBs, the final extract volume will be 10 ml. The Hexane/Acetone extract is concentrated to just under 10 ml. Using a glass pipette, the extract is transferred to a 15 ml vial. The TurboVap cell is then rinsed with Hexane. The rinsate is also transferred to the 15 ml vial. The solvent is then quantitatively brought up to a final volume of 10 ml. The 10 ml volume is measured by comparison to another 15 ml vial containing 10 ml of solvent, which in turn has been measured using a class A volumetric cylinder.

9.14.7.3 When TPH, and DRO are being concentrated, the final extract volume should be 1.0 ml. After the TurboVap has finished concentrating to 0.75 ml, bring the extract to 1.0 ml with the solvent used for extraction using a calibrated syringe and quantitatively transfer to a 2.0 ml target vial. The ESS Lab ID is transcribed to the target vial.

9.14.7.4 When SVOA, and LLPAH are being concentrated, the final extract volume is 0.5 ml.

9.14.8 The extract is now ready to be analyzed for the target analytes using the appropriate determinative technique(s). All extracts must be analyzed within 40 days.

10.0 CALCULATIONS

10.1 Not applicable. See determinative method.

11.0 QUALITY CONTROL

11.1 Before processing any samples, the analyst should demonstrate that all parts of the equipment in contact with the sample and reagents are interference-free. This is accomplished through the analysis of a solid matrix method blank (e.g., Sodium Sulfate). Each time samples are extracted, and when there is a change in reagents, method blank needs to be extracted and analyzed for the compounds of interest. The method blank should be carried through all stages of the sample preparation and measurement.

11.2 Standard quality assurance practices should be used with this method. Field duplicates should be collected to validate the precision of the sampling procedures. A matrix spike/matrix spike duplicate, or matrix spike and duplicate sample analysis, and a laboratory control sample should be prepared and analyzed with each batch of samples prepared by this procedure, unless the determinative method provides other guidance.

11.3 Surrogate and spike standards should be added to samples according to the appropriate determinative method.

12.0 DATA VALIDATION

12.1 Refer to the determinative methods for performance data.

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12.2 Any unusual observations are noted in the prep log.

12.3 All logbooks are routinely reviewed by the laboratory supervisor.

12.4 All non-compliances require a corrective action report. This report must be included with the raw data. Refer to the quality control of the determinative method.

13.0 REFERENCES

1.1 SW846 Method 3546, (Nov 2000) Microwave Extraction.

1.2 NELAC/NELAP Chapter 5, rev 16: July 2002.

1.3 DoD QSM Final Version 3, January 2006.

1.4 ESS Laboratory Policies and Procedures.

14.0 POLLUTION PREVENTION and WASTE MANAGEMENT

1.5 ESS Laboratory's policies on pollution prevention and waste management are covered in SOP 90_0002, Hazardous Waste Contingency and Emergency Response Plan. All employees are trained in the requirements of the SOP.

15.0 DEFINITIONS

15.1 Internal Standard - A pure analyte(s) added to a sample, extract, or standard solution in known amount(s) and used to measure the relative responses of other method analytes and surrogates that are components of the same sample or solution. The internal standard must be an analyte that is not a sample component.

15.2 Surrogate Analyte - A pure analyte(s), which is extremely unlikely to be found in any sample, and which is added to a sample aliquot in known amount(s) before extraction or other processing and is measured with the same procedures used to measure other sample components. The purpose of the SA is to monitor method performance with each sample.

15.3 Laboratory Duplicate – A second aliquot of a sample taken in the laboratory and analyzed separately with identical procedures as the sample. Analyses of the lab duplicate and sample indicates precision associated with laboratory procedures, but not with sample collection, preservation, or storage procedures.

15.4 Field Duplicates - Two separate samples collected at the same time and place under identical circumstances and treated exactly the same throughout field and laboratory procedures. Analyses of the sample and its field duplicate give a measure of the precision associated with sample collection, preservation and storage, as well as with laboratory procedures.

15.5 Method Blank - An aliquot of reagent water or other blank matrix that is treated

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exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The Method Blank is used to determine if method analytes or other interferences are present in the laboratory environment, reagents, or the apparatus.

15.6 Field Blank (FB) - An aliquot of reagent water or other blank matrix that is placed in a sample container in the laboratory and treated as a sample in all respects, including shipment to the sampling site, exposure to sampling site conditions and equipment, storage, preservation, and all analytical procedures. The purpose of the FB is to determine if method analytes or other interferences are present in the field environment.

15.7 Blank Spike (BS) - An aliquot of reagent water or other blank matrix to which known quantities of the method analytes are added in the laboratory. The BS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.

15.8 Matrix Spike (MS) - An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The MS is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS corrected for background concentrations.

15.9 Stock Standard Solution - A concentrated solution containing one or more method analytes prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source.

15.10 Calibration Standard (CAL) - A solution prepared from the primary dilution standard solution or stock standard solutions and the internal standards and surrogate analytes. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

15.11 Quality Control Sample - A solution of method analytes of known concentrations which is used to fortify an aliquot of BS or sample matrix. The quality control sample (known as second source standard) is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check laboratory performance with externally prepared test materials.

16.0 METHOD PERFORMANCE

See determinative methods for method performance information.

17.0 PERSONNEL QUALIFICATIONS

17.1 BA/BS Chemistry, Natural & Applied Science or equivalent experience.

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17.2 All analysts, before performing any analysis, participate in the ESS Laboratory training program (SOP80_0016). The training process consists of reading the Standard Operating Procedure, gaining instruction on the procedure from an experienced analyst, and performing the initial demonstration of capability.

18.0 TROUBLESHOOTING

18.1 If a pressure error message appears during the run, it may be necessary to re-enter the pressure calibration number (PCN) and Serial Number (SN) into the microwave parameters. The PCN is 4273-4271-0542-0542-6634-0261. and the SN is MD9083.

18.2 To enter the PCN:

18.2.1 Press the "Setup" button on the Microwave instrument interface.
18.2.2 Using the arrow keys, highlight " Select Sensor" and then press the "Select" button
18.2.3 Using the arrow keys, highlight " Pressure Sensor" and then press the "Select" button.
18.2.4 Next, highlight " ESP-1500 Plus" and then press the "Select" button.
18.2.5 Next, highlight " Enter Calibration Constant" and then press the "Select" button. Enter the 24 digit PCN number. Press "Select" to finish.

18.3 To enter the SN:

18.3.1 Press the "Setup" button on the Microwave instrument interface.
18.3.2 Using the arrow keys, highlight " System Information" and then press the "Select" button
18.3.3 Using the arrow keys, highlight " Service Information" and then press the "Select" button.
18.3.4 Using the arrow keys, highlight " SN" field and then press the "Select" button. Enter the SN number. Press "Select" to finish.

19.0 DATA MANAGEMENT AND RECORDS

19.1 **Data Management** - ESS Laboratory's utilizes the Promium Element LIMS system as part of its data management system. Client sample information is entered into ELEMENT LIMS and analyses are assigned to each sample. The LIMS allows EPA hold times, minimum batch QC requirements, and QC criteria to be assigned to each analysis. Standards can be entered and assigned to QC samples through the LIMS. Once analysis has been performed, data is imported using DataTool avoiding manual transcription errors. In conjunction with Crystal Reports, the ELEMENT system allows for a wide variety of reporting formats.

19.2 **Records** – The specific retention periods required in the NELAC Standards, EPA-CFR and state and local statutes are followed or exceeded. At a minimum, data records are retained for five years from last use (10 years for drinking water). If there is a question

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about whether a record should be retained or disposed because no specific requirement could be found, the record is retained until such time as a retention period is specified. Records are stored in specified-labeled locations and are easily retrievable. All raw data associated with testing is also retained including; computer printouts, chromatograms, review forms, and logbooks.

20.0 ATTACHMENTS (Including TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA)

20.1 Attachment A – Organic Prep Logbook

20.2 Attachment B – Primary Reagent II Logbook

20.3 Attachment C – Percent Solids Logbook

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Attachment A

Procedure: 50.3546 R. 0
Microwave Extraction
Procedure Document

ESS Organic Preparation Logbook

Project #:_____ Surrogate ID# Matrix Spike ID# Analytical Matrix:_____
Prep Date:_____ A_____ D_____ Extraction Time:
Batch ID:_____ B_____ E_____ Start:_____
Extraction Method:_____ C_____ F_____ Finish:_____

Split Extraction* ☐
* Half of the final extract volume (0.5ml) is exchanged into 5ml 5ml hexane and transferred as Vol 1. The other half (0.5ml CH_2Cl_2) is transferred as Volume 2.

ESS ID	Vol(ml)/ Wt.(g)	Surrogate (ul or ml)	Matrix Spike (ul or ml)	Final Extract Vol (ml) Hex/CH_2Cl_2	Transfer Vol #1 (ml) Hex/CH_2Cl_2	Transfer Vol #2 (ml) Hex/CH_2Cl_2	Transfer Date	Bath Temp (C)	pH	Discard bottle**	Comments	1st Rvw Init.	Witness Init.	2nd Rvw Init.

Analysis Performed
PCB ☐
B/N SVOA ☐
SVOA ☐
LL PAH ☐
PEST ☐
TPH/GC ☐
BIS-2 ☐
PAH ☐

Acid Washed: Y / N
H_2SO_4 ID#
Cu Cleaned: Y / N
Cu ID#
Florisiled: Y / N
Lot#
Silica Column/Carbon prep: Y / N
Lot #

CH_2Cl_2 lot #_____ NaOH ID#_____

Prepared By:_____ Glasswool:_____ Method #(s):_____ Hexane lot#_____ Na_2SO_4 ID#_____

**Check off column if entire sample used and bottle discarded.

Acetone lot#_____

Control #50.0001-0701A BATCH ID/Test:_____ BATCH ID/Test:_____ Page_____

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Primary Reagent Log II

* for Primary Reagents treated in Muffle or Drying Oven

Date	Lab ID #	Reagent Name	1° Lab ID	Preparation			Exp. Date	1st Rvw Init.
				Oven Used	Temp. (°C)	Duration		

IMAGE HERE

Reviewed by:_____ Control #50.0018-0702A Page_____

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ATTACHMENT C

ESS LABORATORY
Percent Solids Logbook

Date/ Time	Lab ID	Pan WT. (g)	Wet WT. (g)	Dry WT. (g)	Percent Solid	Wet wt. Init.	Dry wt./1st Rvw Init.	2nd Rvw Init.

Criteria: Dup RPD ≤ 20%

Control #50.0006-0701A

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ESS Laboratory
Division of Thielsch Engineering
Cranston, RI

SOP NO. 60_8081

ORGANOCHLORINE PESTICIDES BY GAS CHROMATOGRAPHY: CAPILLARY COLUMN TECHNIQUE (EPA METHOD 608/SW-846 METHOD 8081A and B/SM6630 C)

APPROVED BY:

Operations Manager	Date 6/29/10
QA Manager	Date 6/22/10
Laboratory Director	Date 7/8/10

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Organochlorine Pesticides by Gas Chromatography: Capillary Column Technique (EPA METHOD 608/SW-846 METHOD 8081A/SM6630 C)

1.0 SCOPE AND APPLICATION

1.1 This procedure is used to determine the concentrations of various organochlorine pesticides, in extracts from wastewater, ground water, surface water, sludge, soil, and solid matrices. Open-tubular, capillary columns are employed with electron capture detectors (ECD).

1.2 Although performance data is presented for many of the compounds, it is unlikely that all of them could be determined in a single analysis. This limitation results because the chemical and chromatographic behavior of many of these chemicals can result in co-elution. Several cleanup/fractionation schemes are provided in this procedure. Any compound is a potential method interference when it is not a target analyte.

1.3 Compound identification (single component compounds) based on single column analysis should be confirmed on a second column, or should be supported by at least one other qualitative technique. This procedure describes analytical conditions for a second gas chromatographic column that can be used to confirm the measurements made with the primary column. GC/MS is also recommended as a confirmation technique if sensitivity permits.

1.4 This procedure has reporting limits of 0.1 ug/L for aqueous samples and 5 ug/Kg for solid and waste samples. Higher reporting levels will result from reduced sample volumes and/or low percent solids. Lower detection limits are achievable by further concentration of sample extract upon request.

2.0 METHOD SUMMARY

2.1 A measured volume or weight of sample (approximately 1 L for aqueous samples and 2-30 g for soils) is extracted using the appropriate sample extraction technique.

2.2 Liquid samples are extracted at neutral pH with methylene chloride using the separatory funnel technique (SOP 50_3510C). Solid samples are extracted with methylene chloride/acetone using either the automated Soxhlet extraction (SOP 50_3541) or the microwave extraction method (SOP 50_3546). The extracts for liquid and solid samples are exchanged into hexane before analysis.

2.3 A variety of cleanup steps may be applied to the extract, depending on the nature of the co-extracted matrix interferences and the target analytes. After cleanup, the extract is analyzed by injecting a 2-uL sample into a gas chromatograph with a wide-bore fused silica capillary column and electron capture detector (GC/ECD).

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3.0 HEALTH AND SAFETY

3.1 Each employee has been trained and has acknowledged being trained in the safe use and handling of chemicals being used in the laboratory. This training has been performed according to the ESS Training SOP 80_0016 and by the Chemical Hygiene Plan SOP 90_0001 in conjunction with the Safety orientation

3.2 All sample and material handling should be done in a hood while using proper protective equipment to minimize exposure to liquid or vapor. Minimum personnel protective equipment includes the use of laboratory safety glasses, a lab coat or apron, and protective gloves.

3.3 The MSDSs for the concentrated chemicals used in the laboratory are kept on file in a central location that is available for all employees to review.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

4.1 Aqueous samples are collected in 1 Liter borosilicate glass jars with Teflon lined caps. The samples are stored in a dark walk-in cooler at 4° C. Two liters should be provided so samples can be re-extracted when necessary.

4.2 If residual chlorine is present, add sodium thiosulfate.

4.3 Aqueous samples must be extracted within 7 days from date sampled. When analyzing samples for EPA method 608, samples that are not between pH 5-9 ***must*** be extracted within 72 hours.

4.4 Soil/sediment samples are collected in 4–8 ounce jars with Teflon lined caps. The samples are stored in the dark at 4°C. Thirty grams of sample is required for extraction and ten grams is required to determine the percent solids. One hundred grams should be provided so samples can be re-extracted when necessary.

4.5 Soil/Sediment samples must be extracted within 14 days of date sampled.

4.6 All extracts are stored in 10ml Teflon capped vials in the extract storage refrigerator located in the Organic Prep lab. These extracts are stored at 4°C and must be analyzed within 40 days of date extracted.

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

5.1 Sources of interference in this method can be grouped into three broad categories: 1) contaminated solvents, reagents or sample processing hardware; 2) contaminated GC carrier gas, parts, column surfaces or detector surfaces; and 3) the presence of co-eluting compounds in the sample matrix to which the ECD will respond. Interferences co-extracted from the samples will vary considerably from waste to waste. While general cleanup techniques are referenced or provided as part of this

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method, unique samples may require additional cleanup approaches to achieve desired degrees of discrimination and quantitation.

5.2 Interferences by phthalate esters introduced during sample preparation can pose a major problem in pesticide determinations. Common flexible plastics contain varying amounts of phthalate esters, which are easily extracted or leached from such materials during laboratory operations. Cross-contamination of clean glassware routinely occurs when plastics are handled. Avoiding contact with any plastic materials and checking all solvents and reagents for phthalate contamination can best minimize interferences from phthalate esters. Exhaustive cleanup of solvents, reagents, and glassware may be required to eliminate background phthalate ester contamination.

5.3 Glassware must be scrupulously cleaned as soon as possible after use by rinsing with the last solvent used. Detergent washing with hot water, and rinses with tap water should follow this. See SOP 50_0001.

5.4 The presence of elemental sulfur will result in broad peaks that interfere with the detection of early-eluting organochlorine pesticides. Sulfur contamination should be expected with sediment samples. SOP 50_3660 is suggested for removal of sulfur.

5.5 Other halogenated pesticides or industrial chemicals may interfere with the analysis of pesticides. Florisil (SOP 50_3620) cleanup eliminates co-eluting chlorophenols.

6.0 EQUIPMENT/APPARATUS

6.1 **Gas chromatograph**: an analytical system complete with gas chromatograph suitable for on-column and splitless injection and all required accessories including syringes, analytical columns, gases, electron capture detectors (ECD) and DOS based PC system interfaced to the GC with HP Chemstation/ EnviroQuant software.

6.1.1 **Wide-bore Columns**:

6.1.1.1 Column 1 (RTX-CL Pesticide) 30 m x 0.53-mm ID fused silica capillary column, 0.50 um film thickness.

6.1.1.2 Column 2 (RTX-CL Pesticide II) 30m-x 0.53-mm ID fused silica capillary column, 0.42 um film thickness.

6.1.2 **Miscellaneous Instrument Parts**:

6.1.2.1 Single Gooseneck glass liners from either HP or Restek.

6.1.2.2 Gold Seals and Washers (HP 05971-27305)

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6.1.2.3 Graphite Ferrules (0.53mm.)

6.1.2.4 O-rings (HP #5180-4182)

6.1.2.5 HP ceramic tiles for cutting columns.

6.2 **10 uL** glass bore Injector **Syringe**.

6.3 Class A **Volumetric flasks,** various sizes.

6.4 **Sample vials**: glass with Teflon-lined crimp tops, 2.0ml.

6.5 **Hamilton microliter syringes** – 10 µl, 25 µl, 100 µl, 500 µl, and 1000 µl

6.6 **Data system**:

6.6.1 **Computers:** The Semi-Volatiles laboratory has two GC/ECD systems analyzing method 608/8081. SVOA GC7 has an DELL computer with a Windows XP operating system. SVOA GC6 has a DELL computer with Windows 2000 operating system. All computer systems are networked to a Windows 2003 server, which is the destination of all files. A differential back-up is performed nightly and a full back is performed each weekend using Symantec Backup Exec to tapes. As the systems acquires and stores data onto the server, the server becomes full. The data is downloaded and archived onto an external hard drive.

6.6.2 **Software:** HP/Agilent Environmental Chemstation - The software is interfaced to the electron capture detectors and allows the continuous acquisition and storage on machine-readable media of all chromatograms obtained throughout the duration of the instrument program. The software is capable of integrating the abundance in any EICP between specified times. Current versions SVOA ECD3:G1045A version 01.00 and SVOA ECD6:G1701DA Version D.00.01.27.

7.0 REAGENTS AND STANDARDS

7.1 **Reagents**: All reagents should be reagent or pesticide grade for this analysis. Unless otherwise indicated all reagents shall conform to specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

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7.1.1 **Solvents**:

7.1.1.1 **n-Hexane**
7.1.1.2 **Methylene Chloride**
7.1.1.3 **Acetone**
7.1.1.4 **Isooctane (2,2,4-trimethylpentane)**

7.2 **Standards**: Store all standard solutions (stock, working, and matrix spiking), with the exception of primary standards, at 4° C in the dark. Primary standards are stored at room temperature to prevent crystallization and consequent loss of analytes from solution, as recommended by the manufacturer. Studies have determined that there is no noticeable loss of analytes by evaporation during normal storage periods.

7.2.1 **Primary Standards**: Expiration dates of unopened/opened primary standards are as stated in SOP 50.0006 or as stated by the manufacturer, whichever is earlier. Copies of the certificates of analysis are in a binder in the SVOA Dept. These certificates detail the compounds in each of the mixes. Certificates are kept on file in the laboratory. The following stock standard solutions are purchased as certified solutions:

Primary Standard	**Manufacturer**	**Catalog No.**	**Concentration**
Pesticide Mix	AccuStandard	M-8081-SC	1000 µg/ml
Hexachlorobenzene	AccuStandard	APP-9-112	100 µg/ml
PEM Mix	AccuStandard	M-8081-DS	200 µg/ml
TCX/DCB Pest/PCB Surrogate	Accustandard	CLP-032-Rpak	200 mg/L
Toxaphene	Accustandard	P-093S	100 µg/ ml
Chlordane	Accustandard	P-017S	100 µg/ ml
Alachlor Standard	Restek	32204	1000 µg/ ml
Secondary Standard	**Manufacturer**	**Catalog No.**	**Concentration**
Pesticide Second Source	Ultra Scientific	PPM-808C	1000 µg/ml
Hexachlorobenzene	Ultra Scientific	EPA-1125	1000 µg/ml
Toxaphene	Ultra Scientific	PP-270	100 µg/ ml
Chlordane	Ultra Scientific	PP -150	100 µg/ ml
Alachlor Solution	Ultra Scientific	EPA-1068	5000 µg/ ml

7.2.2 **Primary Stock standard** is used to prepare the single-component Pesticide working standards. 100 µl of the primary pesticide mix standard, 500 µl of the primary surrogate standard, 100µl of the Alachlor Standard and 1000 µl of the Hexachlorobenzene standard is added to a 50 ml volumetric flask with the Hamilton syringes and diluted to the 50 ml mark with Hexane. The final concentration for each analyte and surrogate is 2000 ng/ml. ***The standard has an expiration date of one year from preparation or the earliest expiration date of the primary standards (whichever is earliest).***

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7.2.3 **Pesticide Working Standards**

7.2.3.1 **Pesticide Initial Calibration Working Standards** are prepared by dilution of the primary stock standards with Hexane. Standards are prepared in volumetric flasks. Working standards are stored in the 40 ml amber vials for a maximum of six months. The lowest concentration level is at a concentration near but above the method detection limit. The concentrations should correspond to the expected range of concentrations found in real samples and bracket the linear range of the detector. The following table details the concentrations (ng/ml) of each analyte at each individual level.

Target Analyte	Volume Stock (7.2.2)	Initial Conc. (ng/ml)	Final Volume (ml)	Final Concentration
Level 1	62.5 µl	2000	25.0	5 ng/ml
Level 2	125 µl	2000	25.0	10 ng/ml
Level 3	750 µl	2000	50.0	30 ng/ml
Level 4	2500 µl	2000	100.0	50 ng/ml
Level 5	750 µl	2000	25.0	60 ng/ml
Level 6	1000 µl	2000	25.0	80 ng/ml
Level 7	1250 µl	2000	25.0	100 ng/ml

7.2.3.1.1 All levels are prepared using Hamilton syringes and hexane as the diluent. All analytes and surrogates are at the same concentration, see list of analytes in 8.5.8.4. All standards are measured with a 1000 µl graduated syringe unless otherwise noted. All levels are prepared from dilutions of the stock standard.

7.2.3.2 **Pesticide Continuing Calibration Verification Standard (CCV)**: This is the Level 4 standard as prepared in 7.2.3.1. When performing analysis for the Navy or DoD, the concentration of the CCV must be varied throughout the calibration range with at least one CCV at or below the middle of the calibration range.

7.2.4 **Pesticide Second Source Standards (ICV/LCS/MS)**

7.2.4.1 **Pesticide Second Source Stock standard**: This stock standard is prepared at a concentration of 2000 ng/ml. Fifty (50) µl of the second source pesticide mix from Ultra, 50 µl of the Hexachlorobenzene second source, 250 µl of the surrogate mix and 10 µl of the second source Alachlor mix is added to a 25 ml volumetric flask and diluted to the 25 ml mark with Hexane. ***The standard has an expiration date of one year from preparation or the earliest expiration date of the primary standards (whichever is earliest).***

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7.2.4.2 **Pesticide Second Source Initial Calibration Verification (ICV) Working Standard** is prepared at a concentration of 50 ng/ml. 1.25 ml of the second source stock standard (7.2.4.1) is diluted to 50 ml in a 50 ml volumetric flask. The standard is transferred to a 40 ml amber vials.

7.2.5 **Toxaphene and Chlordane Standards:** Separate standards are prepared for each multi-component target analyte.

7.2.5.1 The **Primary Toxaphene Stock Standard** is prepared at a concentration of 5000 ng/ml for Toxaphene and using hexane as the diluent. 500 µl of the primary Toxaphene Mix from Accustandard, measured with a 1.0 ml graduated syringe, and 4 µl of the primary surrogate mix, measured with a 10 µl graduated syringe, is diluted to 10 ml in a 10 ml volumetric flask. (Surrogate at 80 µg/L.)

7.2.5.2 The **Toxaphene Working Standards** are prepared from the Primary Toxaphene stock standard (7.2.5.1). All levels are prepared using a 1000 µl graduated syringe and diluted with hexane.

Level	Initial Conc. (ng/ml)	Amount Added (µl)	Final Vol. (µl)	Final Conc. Toxaphene (ng/ml)	Final Conc. Surrogate (ng/ml)
1	5000	50	1000	250	4
2	5000	100	1000	500	8
3	5000	200	1000	1000	16
4	5000	400	1000	2000	32
5	5000	500	1000	2500	48
6	5000	800	1000	4000	64
7	5000	1000	1000	5000	80

7.2.5.3 DELETED

7.2.5.4 Prepare the **Toxaphene Second Source Working Standard** at 2500 ng/ml by diluting 250 µl of Toxaphene Standard (Ultra Scientific) and 2 µl of surrogate to a final volume of 10 ml, with hexane.

7.2.5.5 The **Chlordane Stock Standard** is prepared at a concentration of 10 µg/ml using hexane as the diluent. 100 µl of the primary Chlordane Mix from Accustandard, measured with a 1000 µl graduated syringe, and 50.0 µl of the primary surrogate mix, measured with a 100 µl graduated syringe, is diluted to 10 ml in a 10 ml volumetric flask. (Surrogate at 1.0 µg/ml.)

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7.2.5.6 The **Chlordane Working Standards** are prepared at five levels from the Chlordane stock standard (7.2.5.3). All levels are prepared using a 1000 µl graduated syringe and diluted with hexane.

Level	Initial Conc. (ppm)	Amount Added	Final Volume (ml)	Final Conc. Chlordane (ng/ml)	Final Concen. Surrogate (ng/ml)
1	10	25 (µl)	10	25	2.5
2	10	50 (µl)	10	50	5
3	10	100 (µl)	10	100	10
4	10	2.5 (ml)	100	250	25
5	10	500 (µl)	10	500	50
6	10	750 (µl)	10	750	75
7	10	1.0 (ml)	10	1000	100

7.2.5.7 **The Chlordane Second Source Standard (ICV)** is prepared at 250 ng/ml using hexane as the diluent. 125 µl of the Ultra Scientific Chlordane Mix measured with a 500 µl graduated syringe and 6.25 µl of the primary surrogate mix, measured with a 25 µl graduated syringe, are diluted to 50 ml in a 50 ml volumetric flask.

7.2.6 **Surrogate/Matrix Spike Solution**: Prepared according to the following chart.

Analyte	**Initial Conc.**	**Amount**	**Final Volume**	**Final Conc.**
TCX/DCB (Surrogate)	200 ug/ml	500 µL[3]	200 ml[1]	0.5 ug/ml
Pesticide Mix (Ultra) Hexachlorobenzene (Ultra)	1000 ug/ml	50 µl[2]	100 ml	0.5 ug/ml
Alachlor (Ultra)	5000 ug/ml	10 µl[4]	100 ml	0.5 ug/ml

[1] Volumetric glassware size (e.g., final vol. of 200 ml made up in a 200 ml vol. flask).
[2] 100 µl syringe is used.
[3] 1000 µl syringe is used.
[4] 25 µl syringe is used.

8.0 PROCEDURE

8.1 Extraction:

8.1.1 Water samples are extracted at a neutral pH with methylene chloride using separatory funnel extraction (SOP 50_3510). Extract solid samples with methylene chloride-acetone (1:1) using automated Soxhlet extraction (SOP 50_3541) or microwave extraction (SOP 50_3546) procedures. ***NOTE: When solids are analyzed for MA MCP or DoD, they must be extracted by the automated Soxhlet (SOP 50.3541) or microwave (SOP 30.3546) methods.***

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8.2 **Cleanup/Fractionation**:

8.2.1 Cleanup procedures may not be necessary for a relatively clean sample matrix, but most extracts from environmental and waste samples will require additional preparation before analysis. The specific cleanup procedure used will depend on the nature of the sample to be analyzed and the data quality objectives for the measurements. General guidance for sample extract cleanup is provided in this section:

8.2.1.1 Perform Florisil Cleanup (SOP 50_3620) to separate pesticide residues from other chlorinated hydrocarbons (polar compounds).

8.2.1.2 Perform Sulfur Cleanup (SOP 50_3660) to remove elemental sulfur, which interferes with electron capture gas chromatography of certain pesticides.

8.2.1.3 Perform Gel Permeation Cleanup (SOP 50_3640) to separate pesticide residues from other high molecular weight hydrocarbons and sulfur compounds.

8.2.1.4 Perform Carboprep Cleanup to remove mid- to high non-polar compounds.

8.3 **Instrument Maintenance**: See Section 19.0 for instrument maintenance and troubleshooting instructions.

8.4 **Instrument Set Up**:

8.4.1 Operating Conditions: Set the chromatographic system to the following operating conditions:

	GC 3 w/single injector	GC 6 w/dual injector
Injector temperature =	200 °C	200 °C
Detector temperature =	300 °C	300 °C
Oven temperature Program:		
Initial temperature =	120 °C	120 °C
Initial time =	1.00 min	1.00 min
Ramp rate =	12.0°C/min to 300°C	9.0°C/min to 300°C
Final time =	9 min	10 min

Carrier gas = helium at 0.99999 purity.

Constant pressure =	2.6 psi	2.8-3 psi

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8.5 **Initial Calibration (single component analytes)**:

8.5.1 ESS Laboratory's policy is that the audit trail on the Chemstation/Enviroquant software is always on. This ensures that any changes made to the instrument operating method be documented through the audit trail.

8.5.2 All acceptance criteria for initial and continuing calibration apply to both the primary and secondary columns.

8.5.3 **Priming the Column**: Once the chromatographic system operating conditions have been established, calibration may begin. Because of the low concentration of pesticide standards injected on a GC/ECD, column adsorption may be a problem when the GC has not been used for a day or more. Therefore, the GC column should be primed or de-activated by injecting a pesticide standard mixture approximately 20 times more concentrated than the mid concentration standard. Inject this standard mixture prior to beginning the initial calibration or calibration verification.

CAUTION: Several analytes, including Aldrin, may be observed in the injection immediately following this system priming. Always run an acceptable solvent blank prior to running any standards or samples.

8.5.4 **Loading the instrument**: All standards and samples are transferred with disposable pipettes into 2 ml target vials designed to fit the HP auto-sampler. The target vials are labeled with the ID of the standard or sample using a fine point marker. The tray on the auto-sampler is numbered 1-99. The instrument can be configured in two ways:

Configuration 1 (GC7): The primary and confirmation columns are connected to glass y-splitter, which is connected to a single injector. Each standard and sample is injected once.

Configuration 2 (GC6): Each column is connected to an independent injection system. Each standard and sample must be injected twice; once on each column. The standards and samples are staggered on the run (may also use glass y-splitter, which is connected to a single injector).

The PEM standard is placed in slot 1. The 7-level standards are placed in slots 2-8. This is the setup for the calibration standards. Vials can be placed in different slots as long as the slot number is written in the logbook.

8.5.5 **Log Book**: All samples set up on the instrument must be entered into the run logbook prior to sample analysis. The logbook must be filled out completely with the date, vial number (slot number), computer file number,

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method number, ESS lab ID, and the initials of the analyst setting up the instrument.

8.5.5.1 Date includes the day, month, and year.

8.5.5.2 Vial number: This field ***must*** be filled in for each entry.

8.5.5.3 Computer file ID is an abbreviation of the path and file associated with a particular vial number. (e.g. vial #3 analyzed on 3/12/98 has a computer file ID of 031298003)

8.5.5.4 The ESS Lab ID includes the ID of the standards, samples and all QC samples.

8.5.5.5 Initials are signed by the analyst setting up the instrument.

8.5.5.6 The Comment section is a summary of calibration results, dilution information, and any unusual observations. (Examples would include: carry over information into the sample, retention time shift, injection time, calibration standard is less than 15% recovery, sample needs dilution.)

8.5.5.7 The Method section is the method in the chromatographic software used to operate the instrument.

8.5.6 At the beginning of each 12-hour shift a **Performance Evaluation Mixture** (PEM) is run to check for degradation problems. The PEM must also be analyzed before the initial calibration. DDT and Endrin easily degrade in the injection port when the injection port or front of the column is dirty. When the PEM is injected, the degradation products of 4,4'- DDT (4,4'-DDE and 4,4'-DDD) are analyzed along with the degradation products of Endrin (Endrin aldehyde and Endrin Ketone). If the degradation of either DDT or Endrin exceeds 15%, take corrective action before proceeding with calibration. Corrective action includes the following:

8.5.6.1 Maintaining the injection port. DDT and Endrin breakdown can be minimized by thoroughly cleaning the injection port. Particulates on the surface of the injection port or liner can contribute to the Endrin breakdown.

8.5.6.2 High Endrin breakdown can also occur while using a y-split, if particles get trapped in the y-split taper replace y-split.

8.5.6.3 A poorly cut column can contribute to the DDT breakdown. Check the column to ensure the end is not jagged.

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8.5.6.4 The percent breakdown is calculated as shown in section 9.4.

8.5.7 After the PEM mix has been analyzed and is within the required QC criteria, the analysis of the 7 level calibration standards may begin. All 7-level standards are injected like the sample using the instrument set-up criteria in section 8.4. The standards are injected from the lowest to the highest standard (Pesticide Level 1 – Level 7).

8.5.8 **Starting a run with Chemstation Software**: The GC is controlled through Chemstation. These methods are set up with the instrument setup information from section 8.4. All of the operating parameters are saved in Chemstation under method file ID. The PT8081 method operates the front and rear injectors. These methods are saved in the C:\HPCHEM\5\METHOD\ directory. A copy of the Chemstation method is in Attachment A. To run the instrument:

8.5.8.1 Open the **Chemstation** icon.

8.5.8.2 From the Chemstation Menu select **Sequence**.

8.5.8.3 In **Initial Setup** type the name of the directory where data will be stored. For the pesticide analysis, all data is stored in a directory called GE for GC3 and GH for GC6 followed by the date. (E.g. if samples were set up to run on 9/12/05 on GC3 then the directory would be called GE091205)

8.5.8.4 In **Analytical Sequence** type in the vial numbers that correspond to when the front and rear injectors will start and stop analyzing.

8.5.8.5 In the **Sample List** type in the ID's of the samples and standards next to the corresponding vial numbers.

8.5.8.6 Select **Save** to save the newly created sequence. Save the sequence with the day created or the ID. (e.g.: A sequence saved on Monday is saved as Mon.s. Sequences do not have to be saved and can be easily recreated. Saving a sequence can save time if for any reason the analytical sequence needs to be restarted due to an instrument or computer malfunction.

8.5.8.7 Start the analysis by selecting **Run Analytical Sequence**.

8.5.8.8 A Chemstation data file will be created for each sample and standard. These files will be stored in the directory C:\HPCHEM\1\data.

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8.5.9 Reviewing the Initial Calibration Data: All GC Chemstation Data is reduced with the EnviroQuant software. (Refer to EnviroQuant Operator's Manual).

8.5.9.1 Setting up an EnviroQuant Method: All pesticide methods are set up as documented in Attachment A. With each new calibration, the response and absolute retention times are updated. The absolute retention times are taken from Pesticide Level 4 (50 ppb).

8.5.10 Updating a New Initial Calibration:

8.5.10.1 Load any pesticide method.

8.5.10.2 Save the method under the date that the standards and samples were set up to run. Methods are saved with the designator 8081 followed by two letters that are sequentially increased for each new calibration generated. (e.g: 8081AA, 8081AB, 8081AC …) ***Note that the ID of this new method is recorded in the Method column of the RUN logbook.***

8.5.10.3 Convert and link all Chemstation data files to EnviroQuant files.

8.5.10.4 Load the Pesticide Level-4 standard file. Go into QEDIT and correctly integrate all of the pesticides. The following is the order of elution for the individual analytes:

Elution Order RTX-CL Pest.	**Elution Order** RTX-CL Pest. II	**Compound**	**Elution Order** RTX-CL Pest.	**Elution Order** RTX-CL Pest. II	**Compound**
1	1	TMX (Surr.)	14	13	Endosulfan I
2	2	Hexachlorobenzene	15	15	Dieldrin
3	3	a-BHC	16	16	Endrin
4	4	g-BHC	17	17	4,4'-DDD
5	5	b-BHC	18	18	Endosulfan II
6	6	d-BHC	19	19	4,4'-DDT
7	7	Heptachlor	20	20	Endrin Aldehyde
8	9	Aldrin	21	22	Methoxychlor
9	8	Alachlor	22	21	Endosulfan Sulfate
10	10	Heptachlor Epoxide	23	23	Endrin Ketone
11	11	g-chlordane	24	24	DCB (Surr.)
12	12	a-chlordane			
13	14	4,4'-DDE			

8.5.10.5 Go into the initial calibration and update the absolute retention times and responses.

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8.5.10.6 For Levels 1,2,3,5, 6, and 7 update only the responses. ***After generating the initial calibration curve in Enviroquant, the analyst must visually check that each calibration standard was entered into the new calibration method. This is accomplished by checking that the area response for one compound from each calibration standard's printout corresponds to the area count listed in the calibration method in Enviroquant***

8.5.10.7 The software is set up to calculate the average response factor from the 7-level curve (Section 9.0). The percent relative standard deviation of the response factor in each of the 7-levels must be less than 20% (Note: the requirement is less than 10% in Method 608). If any of the pesticides are outside of this criterion, check the data setup in the EnviroQuant software and review the integration of each of the 7-levels. If manual integrations need to be made, update responses again.

8.5.10.8 When updating responses, all peaks in each level should fall within the retention time windows established according to section 8.6. If the peaks have drifted outside of the retention time windows, then the initial calibration is not acceptable. This peak drift is usually due to a carrier gas leak or clog in a gas line. Most leaks come from the injection port. To correct a leak, start by tightening all connections, including the column nut, gold seal, and insert retainer nut. If this doesn't work, go to section 19.0 and replace the ferrule, the septa, and the o-ring. In some cases, the gooseneck liner will get clogged with a piece of septa. Inspect the liner for pieces of septa. If the problem persists, consult with supervisor or a service representative. Once the problem has been corrected, reanalyze the entire initial calibration.

8.5.10.9 For those analytes of interest that continue to be greater than 20% RSD, use a linear calibration or non-linear calibration (quadratic regression). The linear calibration is a regression equation that does not pass through zero. See Calculations in section 9.0. The instrument response is treated as the dependent variable (y) and the concentration as the independent variable (x). The regression will produce the slope and intercept terms for a linear equation in the form:

$$y = ax + b$$

Where :

y= instrument response (peak area)
a= Slope
x= Concentration
b= intercept

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8.5.10.9.1 The regression calculation will generate a coefficient of determination that is a measure of the 'accuracy of fit' of the regression line to the data. In order to be used for quantitative purposes, the calibration curve must have a correlation coefficient of $r \geq 0.995$ or coefficient of determination of $r^2 \geq 0.99$. A minimum of five consecutive points are needed for average response or linear regression. A minimum of six consecutive points are needed for quadratic regression. The analyst should select the regression order that introduces the least calibration error into the quantitation. *The maximum allowable %RSD for each target analyte is 40%. Refer to* ***Attachments C and E*** *for further details.*

8.5.10.10 Document all maintenance performed in the instrument log.

8.5.10.11 Once all integrations are correct, the %RSD is less than 20%, $r \geq 0.995$ or $r^2 \geq 0.99$ and all standards are within the RT windows, save the method.

8.5.10.12 After the Initial calibration has been generated, the analyst must create a copy of the 10 ppb standard and quantitate the file with the new calibration. Review of the analyte recoveries in the standard will demonstrate the appropriateness of the calibration curve.

8.5.10.13 Analysis of a second source calibration verification standard is performed immediately after initial calibration (7.2.4.2). Acceptance criterion is 80-120% with no exceptions.

8.6 Retention Time Windows

8.6.1 Retention time windows are central to the identification of target compounds. Absolute retention times are used for compound identification in all GC and HPLC external standard methods. Retention time windows are established to compensate for minor shifts in absolute retention times as a result of sample loadings and normal chromatographic variability. The width of the retention time window should be carefully established to minimize the occurrence of both false positive and false negative results.

8.6.2 Determining Retention Time Windows:

8.6.2.1 Before establishing retention time windows, make sure that the chromatographic system is operating reliably and that the system

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conditions are optimized for the target analytes and surrogates in the sample matrix to be analyzed.

8.6.2.2 Make three injections of the mid-range CCV single component standard mixtures and multi-component analytes (Toxaphene and Chlordane) over the course of a 72-hour period. Serial injections or injections over a period of less than 72 hours may result in retention time windows that are too tight.

8.6.2.3 Record the retention time in minutes for each single component analyte and surrogate to three decimal places. Calculate the mean and standard deviation of the three absolute retention times for each single component analyte and surrogate. For multi-component analytes, calculate the mean and standard deviation of three to five peaks used for calibration or identification.

8.6.2.4 If the standard deviation of the retention times for a target compound is 0.000 (i.e., no difference between the absolute retention times), then use a default standard deviation of 0.01 minutes. The width of the retention time window for each analyte, surrogate, and major constituent in multi-component analytes is defined as ± 3 times the standard deviation of the mean absolute retention time established during the 72-hour period or 0.03 minutes, whichever is greater.

8.6.2.5 Establish the center of the retention time window for each analyte and surrogate by using the absolute retention time for each analyte and surrogate from the mid-range CCV (Level 4 standard) at the beginning of each analytical shift. For samples run during the same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration.

8.6.2.6 Absolute retention time windows must be calculated for each analyte and surrogate on each chromatographic column and instrument used.

8.6.2.7 New retention time windows must be established when a new GC column is installed or when GC column has been shortened during maintenance.

8.6.2.8 Surrogates are added to each sample, blank, QC sample and calibration standard. Surrogate retention times in the calibration standards are useful in tracking retention time shifts. Whenever the observed retention time of a surrogate is outside of the established retention time window for standards, the analyst is advised to determine the cause and correct the problem before continuing analyses.

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8.7 **Sample Analysis**: Once the initial calibration has passed all of the quality control criteria, sample analysis may begin. All samples and standards must be run under the same conditions as the initial calibration.

8.7.1 **Sequence of Analysis:** At the beginning of each 12-hour shift, the PEM must be successfully analyzed. Samples cannot be analyzed more than 12 hours after the first PEM standard is injected and no more than 20 samples can be analyzed within the 12 hour period. Immediately after the PEM and prior to sample analysis the initial calibration must be verified with the mid-range CCV (Level 4 working standard). Samples are immediately analyzed after the CCV. Samples include the method blank, blank spikes, samples, matrix spikes, and matrix spike duplicates. **NOTE: When analyzing for DoD, a CCV must be analyzed after every 10 field samples and at the end of each analytical sequence**. The CCV alternates between the Pesticide Level 2 and 4 standards.

NOTE: 1) ***Analysis of a second source calibration verification standard is performed immediately after initial calibration in place of the CCV.*** 2) ***It is not acceptable practice to group QC samples together and/or to analyze QC samples on one instrument and their associated samples on another instrument. Analyst must try to analyze batch QC samples, as capacity allows, along with their associated field samples.***

8.7.2 **Loading calibration verification standards and samples**: See section 8.5.4.

8.7.3 **Log Book entries**: See section 8.5.5.

8.7.4 **Running Continuing Calibration Standards and Samples with Chemstation Software:** See section 8.5.8

8.7.5 **Reviewing Continuing Calibration Standard and Sample data:** All GC Chemstation data is reduced with the Enviroquant Software. (Refer to Hewlett Packard's EnviroQuant Operators Manual)

8.7.5.1 Update the Method with Continuing Calibration standards:

8.7.5.1.1 Open the Enviroquant Icon on the computer connected to the instrument that ran the samples and standards.

8.7.5.1.2 Load the method with the initial calibration data that corresponds to the samples and standards analyzed. See section 8.5.9.11. Usually this is the last method entered into the logbook.

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8.7.5.1.3 Convert all Chemstation files to Enviroquant Files

8.7.5.1.4 Load the CCV standard that ran just prior to the first sample set up in the analytical sequence. Go into QEDIT and correctly integrate all of the pesticides. Go into the initial calibration and update the absolute retention times only. **Do not update responses!** The order of the pesticide elution for each of the columns is listed in section 8.5.10.4. The CCV standard should only have 22 predominant peaks. If there are more or less than 22 peaks then refer to the following corrective action and reanalyze all standards.

Problem	Corrective Action
Wrong standard injected	Check sequence setup section 8.5.4. Make sure standards are correctly placed on tray.
Contaminated standard	Inject another aliquot of standard or remake standard.
Standard prepared incorrectly	Remake standard
Dirty injection port	Perform maintenance. Refer to section 19.0

8.7.5.1.5 Save the updated method.

8.7.5.2 Reviewing the Continuing Calibration Standards:

8.7.5.2.1 Use the method updated in section 8.7.5.1.5 to analyze (calculate and generate report) the calibration standards which ran before and after the samples of interest.

8.7.5.2.2 If the retention times have shifted outside the retention time window, see section 8.6. If there is high breakdown, perform the maintenance in section 19.0.

8.7.5.2.3 The CCV standards must be injected at intervals of not less than once every 12 hours or 20 samples (10 field samples for DOD) and at the end of the analysis sequence. Note: every 10 samples is recommended to minimize the number of samples requiring re-injection when QC limits are exceeded. Concentration levels must alternate between low and high concentration standards (equivalent to second and fourth levels in calibration curve). See calculations in section 9.0. Acceptance criteria are as follows:

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Routine Analysis	Percent difference/drift $\leq$ 20% for all reported analytes
DoD	-Percent difference/drift $\leq$ 20% for all reported analytes
MCP CAM	-Percent difference/drift $\leq$ 20% for all reported analytes

8.7.5.2.4 The breakdown in the PEM standard must meet criteria in Section 8.5.5. All of the retention times must also be within the established Retention Time windows (see section 8.6). When these criteria are exceeded, inspect the gas chromatographic system to determine the cause and perform whatever maintenance is necessary before verifying calibration and proceeding with sample analysis. (See section 19.0) If routine maintenance does not return the instrument performance to meet the QC requirements based on the last initial calibration, then a new initial calibration must be performed.

8.7.6 Sample injection may continue for as long as the calibration verification standards and standards interspersed within the samples meet the QC requirements. The sequence ends when the standard that follows a set of samples has been injected or when qualitative and/or quantitative QC criteria are exceeded.

8.7.7 When analyzing samples for multiple component pesticides (Chlordane and Toxaphene), any hits must be verified with a mid-level standard analyzed within 12 hours of the sample. The calibration factors of these analytes must be less than 20% difference when compared to the initial calibration. The retention times must also be within the established RT windows. **When analyzing samples for DoD, samples must be bracketed by CCVs.**

8.8 Sample Data Reduction:

8.8.1 All samples bracketed within acceptable calibration standards are analyzed by the same method used to analyze the standards. Through EnviroQuant, a quantitation report and chromatogram is generated for each sample.

8.8.2 The EnviroQuant software will tentatively identify a single component pesticide when a peak from a sample extract falls within the daily retention time window.

8.8.3 The analyst must carefully review the chromatograms, in QEdit, to ensure that all peaks were identified correctly.

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8.8.3.1 Each blank, blank spike, blank spike duplicate, sample, matrix spike, and matrix spike duplicate should have surrogates identified. If the surrogate has not been identified, go into Qedit and integrate the peak in the surrogate retention time window. In some cases, surrogates cannot be identified due to interferences from unknown components that elute at the same time as the surrogate (see section 11.0 for out of criteria corrective action). This must be noted in the case narrative. If the surrogates are outside the retention time window, then the retention times have shifted. If this happens, the sample must be re-analyzed. Oily samples can cause a retention time shift. These samples will need to be diluted at least 1:2 prior to analysis.

8.8.3.2 The analyst must also review the baseline on all the sample and standard chromatograms. A baseline rise will result from contamination of the injection port and column. When the baseline interferes with sample and standard analysis, re-analyze samples and standards. Attempt to isolate the sample or samples that contaminated the system. Perform cleanup procedure or dilute these samples, if possible, before re-analysis.

8.8.3.3 Late eluting compounds can carryover from sample to sample or sample to standard. Large unresolved peaks in a sample are a good indication of carryover. If carryover occurs, first identify the sample that is causing the carryover. Rerun samples with carryover contamination. Run instrument blanks, consisting of clean hexane, after the sample that carried over until carry over is eliminated. In cases when carry over is too great, the sample may need to be cleaned up further or diluted.

8.8.4 The on-column concentration of each analyte in the sample is provided on the quantitation report. The concentration is in ng/ml. The concentration is calculated from the average response factor or line equation from the initial calibration, which ran prior to analysis.

8.8.5 To quantitate a pesticide analyte, the concentration must be greater than or equal to the MDL. If the analyte concentration is below the MDL then it cannot be reported as detected.

8.8.6 If the analyte concentration is above the MDL and below the level 7 concentration standard, then the result is tentative and must be confirmed.

8.8.7 If the analyte concentration is above the level 7 concentration, then dilute the sample to bring the concentration of the analyte to within the level 1 and level 7 range.

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8.8.8 Confirm pesticide by performing the same analysis on a different column. If the sample was initially analyzed on an RTX-CL1 Pesticide column and there was an analyte detected above the method detection limit, the sample must be confirmed on an RTX-CL2 Pesticide Column. The reverse also holds true.

8.8.9 Analyte results are considered confirmed when there is a peak within the retention time window for the analyte on both the primary and confirmation columns **and** both results are above the MDL.

8.8.10 The final result is determined by comparing the initial analysis with the confirmation analysis.

8.8.10.1 When an analyte result is within calibration range, the primary column result is compared to the confirmation result. If the RPD< 40%, then the higher result is reported. If the RPD > 40%, the analyst must evaluate the chromatogram for co-eluting peaks. If no co-elution is detected and all other QC is within criteria, report the higher result. If co-elution was present, the analyst is to report the result without co-elution and an explanation is to be provided in the case narrative.

8.8.10.2 If the confirmation results are at a concentration greater than the level 7 standard, then a dilution must be made. The sample dilution is analyzed. The criterion stated in 8.8.10.1 is applied to the two results.

8.8.10.3 ESS Laboratory will, upon request, report analytes results to the MDL. Analyte results between the MDL and RL have a greater uncertainty in concentration than results within the calibration range and will be J-flagged as estimated. ***This is standard practice for DoD work.***

8.8.11 Identification of mixtures (i.e., Toxaphene and Chlordane) is based on the characteristic "fingerprint" retention time and shape of the indicator peak(s). Quantitation is based on the area under the characteristic peaks as compared to the area under the corresponding calibration peak(s) of the same retention time and shape generated using external calibration procedures.

8.9 **Quantitation of Multiple Component Analytes:**

8.9.1 Multi-component analytes present problems in measurement. Suggestions are offered in the following sections for handing Chlordane and Toxaphene.

8.9.2 Initial calibration for routine analysis will consist of a one point calibration at the mid point of the calibration curve. **When samples are to be analyzed**

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for DoD, then an initial calibration consisting of a minimum of 5 calibration standards must be generated prior to sample analysis. See section 8.5 for acceptance criteria.

8.9.3 Chlordane is a technical mixture of at least 11 major components and 30 or more minor components. *Trans-* and *cis*-Chlordane are the two major components of technical Chlordane. However, the exact percentage of each in the technical material is not completely defined, and is not consistent from batch to batch. *NOTE: ESS Laboratory calibrates for both alpha- and gamma-chlordane when analyzing pesticides. Both compounds are present in technical chlordane. Therefore, if neither compound is present in the sample extract the laboratory has proof that the sample is non-detect for technical chlordane and will not recalibrate the sample file with the technical chlordane method.*

8.9.3.1 The GC pattern of a Chlordane residue may differ considerably from that of the technical standard. Depending on the sample substrate and its history, residues of Chlordane can consist of almost any combination of constituents from the technical Chlordane, plant and/or animal metabolites, and products of degradation caused by exposure to environmental factors such as water and sunlight.

8.9.3.2 Whenever possible, if Chlordane residue does not resemble technical Chlordane, the analyst should quantitate the peaks of alpha - Chlordane, gamma-Chlordane, and Heptachlor separately against the appropriate reference materials, and report the individual residues.

8.9.3.3 When the GC pattern of the residue resembles that of technical Chlordane, the analyst may quantitate Chlordane residues by comparing the total area of the Chlordane chromatogram using 3-5 major peaks. The peaks are quantitated individually and the average concentration is reported as the result.

8.9.4 When the GC patterns resemble Toxaphene, the analyst will quantitate the chromatogram using the total area of the Toxaphene pattern. To measure total area, construct the baseline of Toxaphene in the sample chromatogram between the retention times of the first and last eluting Toxaphene component in the standard. In order to use the total area approach, the pattern in the sample chromatogram must be compared to that of the standard to ensure that all of the major components in the standard are present is the sample. Otherwise, the sample concentration may be significantly underestimated.

9.0 CALCULATIONS

9.1 Calculate pesticide soils as follows:

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for DoD, then an initial calibration consisting of a minimum of 5 calibration standards must be generated prior to sample analysis. See section 8.5 for acceptance criteria.

8.9.3 Chlordane is a technical mixture of at least 11 major components and 30 or more minor components. *Trans-* and *cis*-Chlordane are the two major components of technical Chlordane. However, the exact percentage of each in the technical material is not completely defined, and is not consistent from batch to batch. *NOTE: ESS Laboratory calibrates for both alpha- and gamma-chlordane when analyzing pesticides. Both compounds are present in technical chlordane. Therefore, if neither compound is present in the sample extract the laboratory has proof that the sample is non-detect for technical chlordane and will not recalibrate the sample file with the technical chlordane method.*

8.9.3.1 The GC pattern of a Chlordane residue may differ considerably from that of the technical standard. Depending on the sample substrate and its history, residues of Chlordane can consist of almost any combination of constituents from the technical Chlordane, plant and/or animal metabolites, and products of degradation caused by exposure to environmental factors such as water and sunlight.

8.9.3.2 Whenever possible, if Chlordane residue does not resemble technical Chlordane, the analyst should quantitate the peaks of alpha - Chlordane, gamma-Chlordane, and Heptachlor separately against the appropriate reference materials, and report the individual residues.

8.9.3.3 When the GC pattern of the residue resembles that of technical Chlordane, the analyst may quantitate Chlordane residues by comparing the total area of the Chlordane chromatogram using 3-5 major peaks. The peaks are quantitated individually and the average concentration is reported as the result.

8.9.4 When the GC patterns resemble Toxaphene, the analyst will quantitate the chromatogram using the total area of the Toxaphene pattern. To measure total area, construct the baseline of Toxaphene in the sample chromatogram between the retention times of the first and last eluting Toxaphene component in the standard. In order to use the total area approach, the pattern in the sample chromatogram must be compared to that of the standard to ensure that all of the major components in the standard are present is the sample. Otherwise, the sample concentration may be significantly underestimated.

9.0 CALCULATIONS

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9.1 Calculate pesticide soils as follows:

$$\text{Pesticide Result ug/Kg} = \frac{\text{(Concentration of Peak ppb)(Extract Volume ml)(Dilution)}}{\text{(\% Solid/100)(Weight of Sample Measured g)}}$$

NOTE: For multi-component pesticides (Chlordane and Toxaphene) use average concentration of 3-5 peaks for Chlordane and total area for Toxaphene.

9.2 Calculate pesticide aqueous as follows:

$$\text{Pesticide Result ug/L} = \frac{\text{(Concentration of Peak ppb)(Extract Volume in ml)(Dilution)}}{\text{Volume of Sample in ml}}$$

NOTE: For multi-component pesticides (Chlordane and Toxaphene) use average concentration of 3-5 peaks for Chlordane and total area for Toxaphene.

9.3 Calculate pesticide matrix spike recoveries as follows:

9.3.1 Matrix spike added is calculated as follows:

$$\text{Matrix Spike Added} = \frac{\text{(Concentration of Spike)(Volume of Spike Added)}}{\text{Initial Volume}}$$

NOTE: Initial volume can be in liters or grams. If it is a soil, make sure to multiply initial volume by % solids/100.

9.3.2 Matrix spike result is calculated as follows:

9.3.2.1 (Refer to Section 9.1, 9.2, 9.3 and 9.4.)

9.3.3 Matrix spike % recovery is calculated as follows:

$$\text{\% Recovery} = \frac{\text{Matrix Spike Result – sample result}}{\text{Matrix Spike Added}} \times 100$$

9.4 Calculate percent breakdown as follows (for DDT and Endrin):

$$\text{\% Breakdown for 4,4'-DDT} = \frac{\text{Total DDT Degradation Peak Area (DDE + DDD)}}{\text{Peak Area (DDT + DDE + DDD)}} \times 100\%$$

$$\text{\% Breakdown Endrin} = \frac{\text{sum of degradation peak area (Aldehyde + Ketone)}}{\text{sum of all peak area (Endrin + Aldehyde + Ketone)}} \times 100\%$$

9.5 For surrogate recoveries, % RSD and all other calculations done by the computer's software refer to Hewlett Packard © 1992 printed in USA 11/92 Part No. HP G1032-90020.

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9.6 Calibration Factor (CF)

CF = Peak Area / Mass of Compound Injected

9.7 Percent Relative Standard Deviation:

% RSD = (SD / Average CF) x 100%

Where: SD = standard deviation

9.8 Percent Difference:

% D = ((CFv – CF ave) / CF ave) x 100%

Where:
CFv = Calibration Factor of the verification standard.
CF ave = The mean calibration factor from the initial calibration.

9.9 Percent Drift:

$$\% \text{ Drift} = \frac{\text{Calculated Concentration} - \text{Theoretical Concentration}}{\text{Theoretical Concentration}} \times 100\%$$

10.0 QUALITY ASSURANCE/QUALITY CONTROL

10.1 Immediately after the initial calibration, a second source standard (ICV) is analyzed. This standard is prepared at the Level 4 concentration. The percent recovery of the second source must be 80-120%. If the percent recovery is outside criterion, then corrective action must be taken. Perform instrument maintenance and/or prepare a new second source standard. If the second analysis of the ICV is not within criterion, then a new calibration curve must be generated. **Sample analysis cannot begin until a valid second source has been analyzed.**

10.2 **Accuracy and Precision**: All laboratory personnel must demonstrate initial proficiency for each sample preparation method/matrix that he or she performs. All new employees must successfully demonstrate initial proficiency prior to independently performing analysis on real samples. This must be accomplished by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The initial proficiency results will become part of each employee's training file.

QC Sample Preparation:

Spiking Solution: Four QC samples must be prepared from a spiking solution with the analytes of interest. The spiking solution must be made using standards

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prepared independently from those used for calibration. The samples must be prepared at a concentration that would result in data falling within the middle of the calibration curve. In most cases the blank spike or matrix spike solution is used. Prep: The samples are prepared in a clean matrix. In most cases this initial demonstration will simply be a matter of preparing four blank spikes with a batch of samples.

QC Sample Analysis: The four QC samples must be analyzed within the criteria of the method being evaluated. The QC samples must be handled in exactly the same manner as actual samples.

Accuracy Calculation: Accuracy is defined as the closeness of agreement between an observed value and an accepted reference value. Each of the four spiked samples will be calculated for percent recovery. The average of the percent recovery values is the accuracy result.

Precision Calculation: Precision is defined as the agreement of a set of replicate measurements without assumption of knowledge of the true value. Precision is estimated by the relative standard deviation (RSD) of the four QC samples.

$\%RSD = (s / x)\ 100\ \%$

Where:
s = Standard Deviation of a finite number of values. On a scientific calculator use the **σxn-1** key.

$\bar{x}$ = The average of the four QC sample % recoveries.

Reporting Accuracy and Precision: Report Accuracy and Precision data with the following minimum info:

Matrix: Prep Method:
Clean-up Method: Analysis Method:
Date Extracted: Date Analyzed:
Sample Prepared by: If Applicable
Sample Fractionated by: If Applicable Sample Analyzed by:
Accuracy: Precision:

Parameter	*% Rec. QC 1*	*% Rec. QC 2*	*% Rec. QC 3*	*% Rec. QC 4*	*Average Recovery*	*Standard Deviation*	*%RSD*

Interpretation of Results: The percent recoveries should be between 50-130% and the %RSD should be less than 30%. If any of the accuracy and precision results do not fall within the criteria then re-prep and re-analyze all QC samples only for those analytes that were not within criteria.

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10.3 A Method Blank is extracted with each 20 samples or analytical batch. The concentration of analytes in the method blank should be less than ½ MRL (MRL is the Level 1 standard). If the concentration of any analyte exceeds ½ MRL, then proceed to Section 11.0 for corrective action.

10.4 Run a 7-point initial calibration curve, using the primary source standards each time major instrument maintenance occurs, or if the CCV does not meet acceptance criteria. Acceptance criteria are ≤ 20%.

10.5 A Blank spike and Blank spike duplicate (BS/BSD) are analyzed with each batch of 20 or less samples. The acceptance criterion for the BS/BSD is 40-140% Recovery, 20% RPD for waters and 30% RPD of the certified value for soils. ***See Attachment A for DoD and blank spike acceptance criteria and see Attachment E for MADEP MCP CAM requirements.*** If the BS/BSD is not within limits, see section 11.0 for corrective action. Sporadic Marginal Exceedance number is as follows:

Number of Analytes in BS	Allowable number of Marginal Exceedances
>90	5
71-90	4
51-70	3
31-50	2
11-30	1
<11	0

Exceedances should be within marginal exceedance limits (Attachment A) or expanded criterion listed above.

10.6 Calculate surrogate standard recovery on all samples, blanks, and spikes. Determine if the recovery is within limits of 30-150%. Both surrogates on both columns must be within criterion. ***See Attachment A for DoD surrogate acceptance criteria and see Attachment E for MADEP MCP CAM requirements.***

10.6.1 If the same surrogate is outside limits on both columns, re-extract the sample.

10.6.2 If both surrogates are outside limits on one column only, then re-analyze the sample extract.

10.6.3 If a surrogate is diluted below the lowest calibration standard, no corrective action is necessary.

10.6.4 If re-extraction or reanalysis yields similar results, the laboratory must note this in the project narrative.

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10.6.5 If re-extraction or reanalysis is performed within hold time and produce results within criterion, then only results from re-extraction or reanalysis needs to be reported.

10.6.6 For MADEP and DoD, if re-extraction or reanalysis is performed outside hold time and produce results within criterion, then both results from re-extraction or reanalysis should be reported.

10.6.7 Note all exceedances in project narrative.

10.7 Continuing calibration verification (CCV) must be performed at the beginning and end of each analytical sequence, see 8.7.5.2.3 for frequency/level of CCV. The CCV uses the Pesticide Level 2 and 4 standards, alternated throughout the analytical sequence.

Acceptance criteria is a follows:

Routine Analysis	Percent difference/drift $\leq$ 20% for all reported analytes
DoD	-Percent difference/drift $\leq$ 20% for all reported analytes
MCP CAM	-Percent difference/drift $\leq$ 20% for all reported analytes

10.7.1 When a CCV is out of this acceptance window, the laboratory should stop analyses and take the following corrective action:

10.7.1.1 When criteria are exceeded then remake and re-analyze CCV If corrective action does not produce a second consecutive (immediate) CCV within acceptance limits, then the analyst may demonstrate the initial calibration is valid by analyzing two consecutive CCVs at two concentrations. If CCV criteria are not acceptable, then a new initial calibration must be performed. ***All samples analyzed after the last acceptable CCV must be re-analyzed except for CCVs that are exceeded high, samples with results that are non-detect may be reported. It has been shown that results would have been detected***

10.7.1.2 When performing analysis for MA MCP, or DoD, any analyte outside of criteria in the CCV must be noted in the project narrative

10.8 The breakdown of DDT and Endrin must be measured before standards and samples are analyzed at the beginning of each 12-hour shift. Injector maintenance

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and re-calibration must be performed if the breakdown is greater than 15% for either compound (CAM, DoD) or greater than 20% combined (DoD). See section 8.5.6 for details. For calculation refer to Section 9.0.

10.9 A matrix spike and matrix spike duplicate (MS/MSD) are analyzed with every batch of 20 samples. The acceptance limits 30-150% Recovery and ± 30% RPD. ***See Attachment A for DoD MS/MSD acceptance criteria and Attachment E for MADEP MCP CAM requirements.*** If the blank spike (BS/BSD) results are acceptable and the matrix spike/matrix spike duplicate results are outside of QC limits, note exception in case narrative.

10.10 Control charts are generated quarterly for the BS (Soil and Water)

10.11 MDLs are determined in reagent water and Ottawa sand/sodium sulfate and verified annually. See SOP 110_0013 for complete MDL study instructions. (Project-specific requirements may require that the MDL study be performed in the site-specific matrix.).

11.0 DATA VALIDATION

11.1 Data validation will be accomplished by reviewing all of the quality control parameters and assuring that they are within recommended ranges by completing the SVOA Analysis Map (see Section 11.3) for GC/ECD Chlorinated Pesticides. The only exceptions made to ranges would be the following:

11.1.1 For MS/MSD, the RPD should be ±30% (***see Attachment A for DoD and Attachment E for MADEP MCP CAM requirements***). However, there are cases where duplicates may not work. If this is the case, inform client in narrative concerning sample non-homogeneity.

11.1.2 For matrix spikes, the % Recovery should be 30-150% ***(see Attachment A for DoD and Attachment E for MADEP MCP CAM requirements)***. If the matrix spike is outside limits, check the BS/BSD. If the BS/BSD is within limits, matrix interferences are present and should be noted in the narrative.

11.1.3 Analytical batches with Method blanks above the MRL (½ the MRL for DoD work) will be re-prepped and re-analyzed with the following exceptions:

11.1.3.1 Samples that are at least twenty times higher than the method blank may be reported.

11.1.3.2 When the method blank is less than 5% of the regulatory limit associated with the analyte the method blank would be acceptable.

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11.1.3.3 If the analyte is found in the method blank above the MRL (½ the MRL for DoD work) but is not in any of the associated samples, no corrective action is needed.

11.1.3.4 Any results that are reported with method blank contamination must be B-flagged.

11.1.4 For the BS/BSD, the %Recovery should be 40-140% ***(see Attachment A for DoD and Attachment E for MADEP MCP CAM requirements***). If the BS/BSD is outside this criteria, the analytical batch will be re-extracted and re-analyzed with the following exceptions:

11.1.4.1 For BS/BSD >140% ***(see Attachment A for DoD and Attachment E for MADEP MCP CAM requirements)***, samples with results below the MRL may be reported. It has been shown that the results above MRL would have been detected.

11.1.4.2 For BS/BSDs < 40% (re-extract for DoD), samples with results above a regulatory limit may be reported.

11.1.4.3 In some instances there may be insufficient sample to re-extract. The client is to be contacted and the results, if reported, are to be reported as estimated values when this occurs.

11.1.4.4 Any samples that are reported with invalid BS/BSD data must have a notation in the case narrative.

11.2 All unusual observations and method deviations will be noted in the narrative accompanying the data report presented to the client.

11.3 All data is reviewed for accuracy by a second analyst. Results of this review are noted on the SVOA Analysis Map in the last column and in the comment section.

12.0 REFERENCES

12.1 SW846, Third Edition, Updates III and IV, Methods 8081A and B. .

12.2 HP GC EnviroQuant User's Guide, HPG1045A

12.3 HP Environmental Data Analysis User's Guide HPG0032C

12.4 HP 6890 GC and HP 5890 Series II Operations Manuals

12.5 Standard Methods, 19th/20th Editions, Method No 6630 C.

12.6 Massachusetts DEP WSC-CAM V B.

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12.7 DoD QSM, Final Version 4.2, October 2010.

12.8 NELAC Standards, Chapter 5, June 2003

13.0 ATTACHMENTS

13.1 Attachment A – DoD QSM Appendix D, QA requirements.

13.2 Attachment B – SVOA Analysis Map.

13.3 Attachment C – Summary of Method Quality Objectives (DOD QSM v4.2).

13.4 Attachment D -- Chemstation method.

13.5 Attachment E – Specific QC Requirements for Mass MCP WSC-CAM-V B.

14.0 POLLUTION PREVENTION and WASTE MANAGEMENT

14.1 ESS Laboratory's policies on pollution prevention and waste management are covered in SOP 90_0002, Hazardous Waste Contingency and Emergency Response Plan. All employees are trained in the requirements of the SOP.

15.0 METHOD PERFORMANCE

15.1 Precision and Accuracy data must be generated by all employees before performing this analysis on client samples. The data is generated by analyzing a method blank and four blank spike samples. Acceptance criteria are 50-130% Recovery and %RSD of ≤ 30%.

15.2 The precision and accuracy data in Table 1 were developed using the Soxtherm extraction method. Values are in ug/L.

16.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

Table 1. Typical Precision and Accuracy data generated 6/1/2005

Compound	Spk	Avg	%Rec	%RSD	Compound	Spk	Avg	%Rec	%RSD
4,4'-DDD	50	55.177	110.4	5.7	Endosulfan Sulfate	50	52.684	116.6	5.6
4,4'-DDE	50	56.231	112.5	6.5	Endrin Aldehyde	50	51.402	105.4	4.5
4,4'-DDT	50	62.526	125.1	6.0	Endrin Ketone	50	52.191	113.7	6.9
alpha-BHC	50	50.965	106.6	7.6	gamma-BHC (Lindane)	50	52.584	102.8	5.4
alpha-Chlordane	50	60.291	101.9	9.4	gamma-Chlordane	50	58.635	104.4	4.4
beta-BHC (1)	50	53.577	120.6	6.5	Heptachlor	50	54.870	105.2	8.5
Decachlorobiphenyl	50	34.056	107.2	6.2	Heptachlor Epoxide	50	54.761	117.3	7.2
delta-BHC	50	52.437	68.1	3.1	Hexachlorobenzene	50	47.598	109.7	7.8
Dieldrin	50	57.653	104.9	6.7	Methoxychlor	50	59.114	109.5	7.0

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Endosulfan I	50	58.857	115.3	6.3	Tetrachloro-m-xylen	50	41.102	95.2	7.9
Endosulfan II	50	58.316	117.7	7.3					

17.0 DEFINITIONS

17.1 **Accuracy**: The closeness of agreement between an observed value and an accepted reference value. When applied to a set of observed values, accuracy will be a combination of a random component and of a common systematic error (or bias) component.

17.2 **Batch**: A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 20, then each group of 20 samples or less will all be handled as a separate batch.

17.3 **Bias**: The deviation due to matrix effects of the measured value (x_s - x_u) from a known spiked amount, where x_s is the spiked sample and x_u is the un-spiked sample. Bias can be assessed by comparing a measured value to an accepted reference value in a sample of known concentration or by determining the recovery of a known amount of contaminant spiked into a sample (matrix spike).

17.4 **Control Sample**: A QC sample introduced into a process to monitor the performance of the system.

17.5 **Equipment Blank**: A sample of analyte-free media which has been used to rinse the sampling equipment. It is collected after completion of decontamination and prior to sampling. This blank is useful in documenting adequate decontamination of sampling equipment.

17.6 **Method Reporting Limit**: The lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The MRL is generally 5 to 10 times the MDL. ESS Laboratory sets the MRL to the lowest non-zero standard in the calibration curve or higher.

17.7 **Field Duplicates**: Independent samples which are collected as close as possible to the same point in space and time. They are two separate samples taken from the same source, stored in separate containers, and analyzed independently. These duplicates are useful in documenting the precision of the sampling process.

17.8 **Blank Spike (BS)**: A known matrix spiked with compound(s) representative of the target analytes. This is used to document laboratory performance.

17.9 **Matrix**: The component or substrate (e.g., surface water, drinking water) which contains the analyte of interest.

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17.10 **Matrix Duplicate**: An intralaboratory split sample which is used to document the precision of a method in a given sample matrix.

17.11 **Matrix Spike**: An aliquot of sample spiked with a known concentration of target analyte(s). The spiking occurs prior to sample preparation and analysis. A matrix spike is used to document the bias of a method in a given sample matrix.

17.12 **Matrix Spike Duplicates**: Intralaboratory split samples spiked with identical concentrations of target analyte(s). The spiking occurs prior to sample preparation and analysis. They are used to document the precision and bias of a method in a given sample matrix.

17.13 **Method Blank**: An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank should be carried through the complete sample preparation and analytical procedure. The method blank is used to document contamination resulting from the analytical process.

17.14 **Method Detection Limit (MDL)**: The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix type containing the analyte. See SOP 110_0013 for further explanation.

17.15 **Organic-Free Reagent Water**: All references to water in the method refer to water in which an interferant is not observed at the method detection limit of the compounds of interest. A water purification system is used to generate organic-free deionized water.

17.16 **Surrogate**: An organic compound which is similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which is not normally found in environmental samples.

18.0 PERSONNEL QUALIFICATIONS

18.1 Analysts who perform this analysis must have a working knowledge of quantitative and qualitative analysis, instrumental methods of analysis, chemical laboratory methods, and equipment.

18.2 All analysts, before performing any analysis, participate in the ESS Laboratory training program (SOP80_0016). The training process consists of reading the Standard Operating Procedure, gaining instruction on the procedure from an experienced analyst, and performing the initial demonstration of capability.

19.0 TROUBLESHOOTING

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19.1 **Instrument Maintenance**: The following procedure is performed when the instrument is initially set up or when a continuing calibration has failed the QC criteria.

19.1.1 Set the GC system to room temperature.

19.1.2 Turn off oven.

19.1.3 Remove column by unscrewing the column in the injection port.

19.1.4 Remove septum nut and septa. Discard septa.

19.1.5 Remove the insert retainer nut. This will expose the O-ring and glass liner. Using a set of tweezers, remove O-ring and liner. If O-ring is not distorted then set aside for later use. Otherwise, replace O-ring. Remove the glass liner. Rinse liner with methanol and scrub with a cotton swab. If the liner is visibly stained, then replace with a new one.

19.1.6 With cotton swab dipped in methanol, clean the injection port and retainer nut.

19.1.7 Remove the gold seal nut located on the bottom of the injection port. With a cotton swab and methanol, clean the gold seal.

19.1.8 Replace all parts in the following order:

19.1.8.1 Gold seal nut. Hand tighten and 1/4 turn with wrench.

19.1.8.2 Insert clean or new glass liner.

19.1.8.3 Place O-ring over liner. Slide O-ring over and down the liner until it fits snug against the injection port.

19.1.8.4 Replace insert retainer nut.

19.1.8.5 Place a new green septa into insert retainer nut.

19.1.8.6 Replace septum nut. **Only hand tighten!**

19.1.8.7 Slide column nut and a new graphite ferrule over column.

19.1.8.8 Using a ceramic tile, cut 3-6 inches off the column. The cut must be square with no jagged edges.

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19.1.8.9 Connect column to injection port by inserting 3 mm of column into the injection port and hand tighten column nut then adding 1/4 turn with a wrench.

19.1.9 Make sure all gases are flowing. (Measure flows with bubble meter.) The flow should be between 5 and 6 ml/min.

19.1.10 Turn on injection port temperature.

19.1.11 Set oven temperature to 120 °C and allow the system to stabilize. Bake out the oven at 300°C for two hours. Reset back to 120°C.

19.2 Record all maintenance in the instrument's maintenance logbook.

20.0 DATA MANAGEMENT AND RECORDS

20.1 **Data Management** - ESS Laboratory utilizes the Promium Element LIMS system as part of its Data Management system. Client sample information is entered into ELEMENT LIMS and analyses are assigned to each sample. The LIMS allows EPA hold times, minimum batch QC requirements, and QC criteria to be assigned to each analysis. Standards can be entered and assigned to QC samples through the LIMS. Once analysis has been performed, data is imported using DataTool avoiding manual errors. In conjunction with Crystal Reports, the ELEMENT system allows for a wide variety of reporting formats.

20.2 **Records** – The specific retention periods required in the NELAC Standards, EPA-CFR and state and local statutes are followed or exceeded. At a minimum, data records are retained for five years from last use (10 years for drinking water). If there is a question about whether a record should be retained or disposed because no specific requirement could be found, the record is retained until such time as a retention period is specified. Records are stored in specified-labeled locations and are easily retrievable. All raw data associated with testing is also retained including; computer printouts, chromatograms, review forms, and logbooks.

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Table 2 DoD Quality Systems Manual Blank Spike QC Limits **(ATTACHMENT A)**

	DoD			
Aqueous				
Analyte	LCL	UCL	LMEL	UMEL
Alpha- BHC	60	130	50	140
Beta- BHC	65	125	55	135
Delta- BHC	45	135	30	150
gamma- BHC (Lindane)	25	135	10	155
Alpha-Chlordane	65	125	55	135
Gamma-Chlordane	60	125	50	135
4,4'-DDD	25	150	10	170
4,4'-DDE	35	140	15	160
4,4'-DDT	45	140	30	155
Aldrin	25	140	10	155
Dieldrin	60	130	50	140
Endolsulfan I	50	110	40	120
Endolsulfan II	30	130	10	150
Endolsulfan Sulfate	55	135	40	150
Endrin	55	135	45	145
Endrin Aldehyde	55	135	40	150
Endrin Ketone	75	125	70	135
Heptachlor	40	130	30	145
Heptachlor Epoxide	60	130	50	140
Methoxychlor	55	150	40	165
Toxaphene	-	-	-	-
TCMX	25	140	-	-
DCB	30	135	-	-
Solids				
Alpha- BHC	60	125	50	135
Beta- BHC	60	125	50	135
Delta- BHC	55	130	45	145
gamma- BHC (Lindane)	60	125	50	135
Alpha-Chlordane	65	120	55	130
Gamma-Chlordane	65	125	55	135
4,4'-DDD	30	135	10	155
4,4'-DDE	70	125	60	135
4,4'-DDT	45	140	30	155
Aldrin	45	140	30	155
Dieldrin	65	125	55	135
Endolsulfan I	15	135	10	155
Endolsulfan II	35	140	20	160
Endolsulfan Sulfate	60	135	50	145
Endrin	60	135	50	145
Endrin Aldehyde	35	145	20	165
Endrin Ketone	65	135	55	145
Heptachlor	50	140	35	155
Heptachlor Epoxide	65	130	55	140
Methoxychlor	55	145	45	155
Toxaphene	-	-	-	-
TCMX	70	125	-	-
DCB	55	130	-	-

%RPD for DoD is ≤30%.

LMEL = Lower marginal exceedance limit UMEL = Upper marginal exceedance limit

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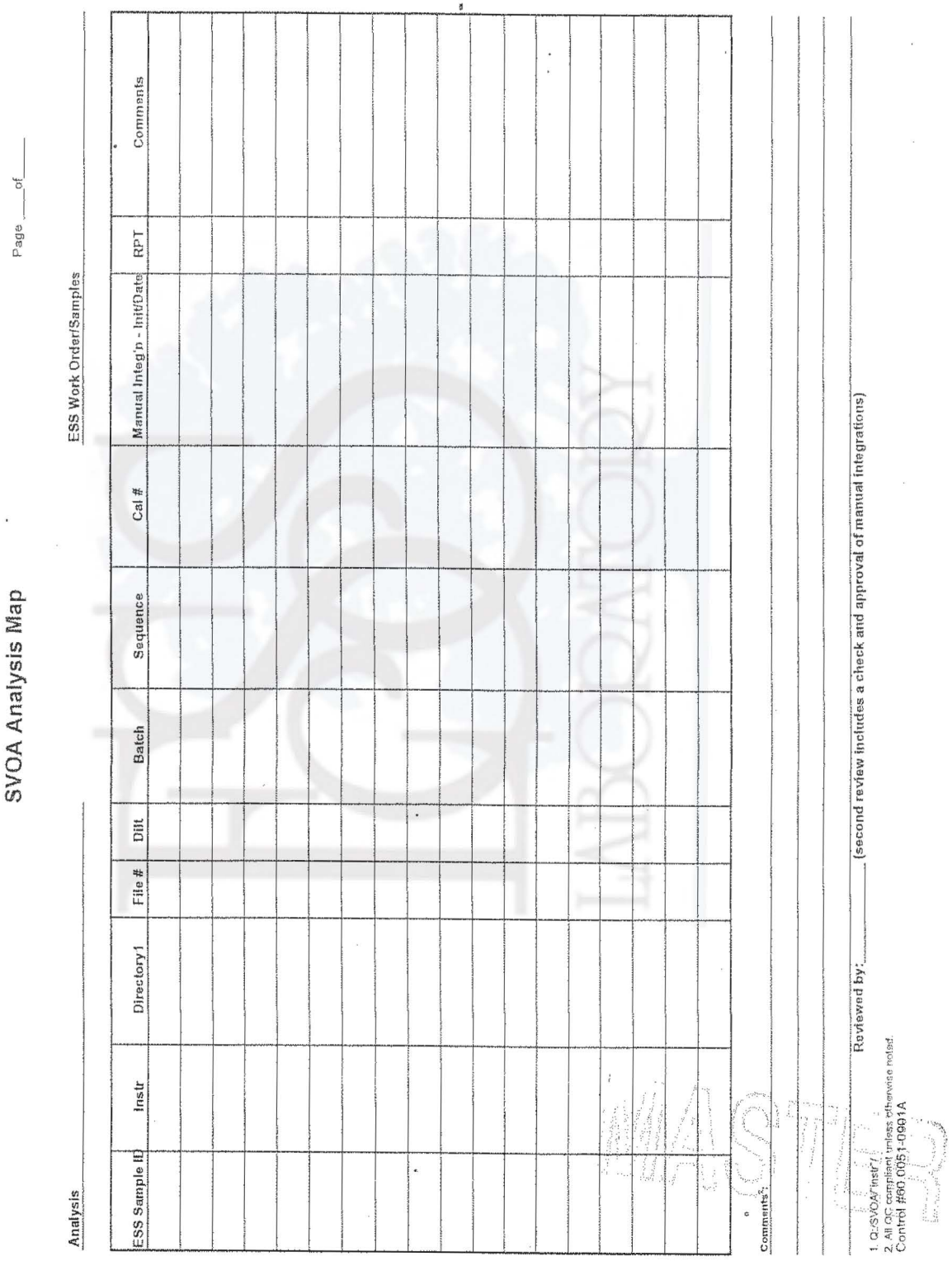
ATTACHMENT B - SVOA Analysis Map



SVOA Analysis Map

Page ___of___

Analysis _____ ESS Work Order/Samples _____

ESS Sample ID	Instr	Directory1	File #	Dilt	Batch	Sequence	Cal #	Manual Integ'n - Init/Date	RPT	Comments

Comments: _____

Reviewed by: _____ (second review includes a check and approval of manual integrations)

1. Q:\SVOA*instr*\
2. All QC compliant unless otherwise noted.
Control #90.0051-0901A

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Table 3 – ATTACHMENT C

Summary of Method Quality Objectives for Method 8081
Chlorinated Pesticide Organic

QC Element	Frequency	Criteria	Corrective Action
PEM – Endrin/DDT Breakdown	Every 12 hours before analysis of standards and samples.	• %Breakdown must be ≤ 15 for Endrin and DDT • Evaluate using peak areas.	• Perform injection port maintenance until successful PEM is obtained
Initial Calibration	Instrument set up. Each time the ICV or CCV cannot meet criteria.	• Minimum of 5 standards and contains all analytes • Low standard ≤ MRL • RSD≤20%, r≥0.995,r^2≥0.99 (Do not force through zero for LR). See section 8 for extended criteria. • For multi-component analytes: Analysis of a single standard at expected mid-point of calibration range. 5 point calibration for ***DOD***	• No allowance. Perform maintenance and recalibrate.
ICV – second source verification standard	Immediately following initial calibration.	• %Rec = 80-120% • Must contain all target analytes. • No allowances	• If criteria are exceeded then remake and re-analyze ICV. If second consecutive ICV is within acceptable criteria then calibration is accepted, otherwise recalibrate. • Report exceedance in narrative
CCV	Prior to sample analysis, every 12 hours and every 20 samples (10 field samples for DoD) and at the end of each analytical sequence.	• Concentration level near midpoint of curve • Must contain all target analytes. • Percent difference or percent drift must be ≤ 20% • Concentration levels must alternate between low and high concentration standards (equivalent to second and fourth levels in calibration curve).	• If criteria are exceeded then remake and re-analyze CCV. If second consecutive CCV is within criteria then calibration is verified, otherwise re-calibrate system and re-analyze any sample analyzed after invalid CCV. ***Exception: If CCV is exhibiting high bias (concentration is higher than upper limit) then any samples that are non-detect for that analyte may be reported.***

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Method Blank	One per analytical batch of 20 or fewer samples.	• Matrix specific • Analytes < MRL (< ½ MRL for DoD work) • DOD also allows < 1/10th the amount measured in the sample or 1/10th the regulatory limit, whichever is greater	• Report exceedance in the project narrative. • Any samples that are non-detect for that analyte may be reported. • Samples with concentrations that are 20x higher than the method blank may be reported. • Samples reported with a contaminated blank must be "B" flagged. • Re-extract if the above exceptions do not apply. If re-extract is within hold, report just the re-extracted data. If re-extract is outside hold then report both sets of data to client.
Blank spike/ Blank spike duplicate	One per analytical batch of 20 or fewer samples.	• Prepared using standard source different than used for initial calibration • Concentration level should be between low and mid-level standard • Must contain all single component analytes • Matrix specific • Percent recoveries between 40-140% (See Table 2 for DoD). • Laboratories must develop in-house limits that are within above criteria.	• Report exceedance in the project narrative. • Re-extract if the above exceptions do not apply. If re-extract is within hold, report just the re-extracted data. If re-extract is outside hold then report both sets of data to client.
Matrix Spike/ Matrix Spike duplicate	One per analytical batch of 20 or fewer samples	• Prepared using the same source as the blank spike • Concentration between low and mid-level standard • Must contain all single component analytes • Matrix specific • Recoveries between 30-150%. ***See Table 2 for DoD.*** • RPD should be < 30% for single component analytes and < 50% for multi-component analytes.	• Check BS/BSD, if recoveries are acceptable then note exceedance in project narrative.

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Surrogates	Added to all samples and standards.	• Use a minimum of 2 surrogates. • Percent recovery of 30-150% *(For USACE work BS/BSD and MB criterion is 50-130% and sample matrix is 40-140%).* ***See Table 2 for DoD. criteria*** • MCP requires that both columns and both surrogates be within criterion and reported. *See Attachment E*	• If the same surrogate is outside of limits on both columns, re-extract samples. If both surrogates are outside criteria on one column only, re-analyze extract • If a surrogate is diluted to a concentration below the lowest standard, then no corrective action is needed. • If surrogates are outside criteria for re-extract, note in project narrative. • If re-extract is within hold and within criteria, report just the re-extracted data. If re-extract is outside hold then report both sets of data to client. • Note exceedance in project narrative. If sample is not re-analyzed due to obvious interference (e.g., UCM), the chromatogram is to be included in the final report.
Identification and Quantitation		• Secondary column analysis: Laboratory must utilize a second dissimilar column to confirm positive pesticide results. • The lab must report the higher of the two results. • All QA/QC requirements must be met on secondary column as well. • Analytes must fall within retention time windows.	• Flag data that is >40% RPD between two columns. • IF high RPD can be attributed to interference on one of the two columns, the lab should report the lower value and provide a discussion in the case narrative that this approach was employed.

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TOPLEVEL PARAMETERS Present GC-6

ATTACHMENT D

Method Information For: C:\MSDCHEM\1\METHODS\8081AQ.M

Method Sections To Run:

(X) Save Copy of Method With Data
( ) Pre-Run Cmd/Macro =
(X) Data Acquisition
(X) Data Analysis
( ) Post-Run Cmd/Macro =

Method Comments:
This is the default method

END OF TOPLEVEL PARAMETERS

INSTRUMENT CONTROL PARAMETERS

Sample Inlet: GC
Injection Source: GC ALS

6890 GC METHOD

OVEN
Initial temp: 120 'C (On)
Initial time: 1.00 min
Maximum temp: 450 'C
Equilibration time: 0.50 min
Ramps:

#	Rate	Final temp	Final time
1	9.00	300	10.00
2	0.0(Off)		

Post temp: 0 'C
Post time: 0.00 min
Run time: 31.00 min

FRONT INLET (SPLIT/SPLITLESS)
Mode: Pulsed Splitless
Initial temp: 200 'C (On)
Pressure: 2.83 psi (On)
Pulse pressure: 15.0 psi
Pulse time: 0.30 min
Purge flow: 30.0 mL/min
Purge time: 0.50 min
Total flow: 35.5 mL/min
Gas saver: Off
Gas type: Helium

BACK INLET (SPLIT/SPLITLESS)
Mode: Pulsed Splitless
Initial temp: 200 'C (On)
Pressure: 3.00 psi (On)
Pulse pressure: 15.0 psi
Pulse time: 0.30 min
Purge flow: 30.0 mL/min
Purge time: 0.50 min
Total flow: 35.7 mL/min
Gas saver: Off
Gas type: Helium

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COLUMN 1
Capillary Column
Model Number: RESTEK 11140
clp pest .53
Max temperature: 330 'C
Nominal length: 30.0 m
Nominal diameter: 530.00 um
Nominal film thickness: 0.50 um
Mode: constant pressure
Pressure: 2.83 psi
Nominal initial flow: 2.7 mL/min
Average velocity: 24 cm/sec
Inlet: Front Inlet
Outlet: Front Detector
Outlet pressure: ambient

FRONT DETECTOR (μECD)
Temperature: 300 'C (On)
Mode: Constant makeup flow
Makeup flow: 60.0 mL/min (On)
Makeup Gas Type: Nitrogen
Electrometer: On

SIGNAL 1
Data rate: 20 Hz
Type: front detector
Save Data: On
Start Save Time: 3.00 min
Stop Save Time: 30.00 min
Zero: 0.0 (Off)
Range: 0
Fast Peaks: Off
Attenuation: 0

COLUMN COMP 1
Derive from front detector

COLUMN 2
Capillary Column
Model Number: RESTEK 11340
RESTEK CLPESTICIDE II 0.53
Max temperature: 330 'C
Nominal length: 30.0 m
Nominal diameter: 530.00 um
Nominal film thickness: 0.42 um
Mode: constant pressure
Pressure: 3.00 psi
Nominal initial flow: 2.8 mL/min
Average velocity: 26 cm/sec
Inlet: Back Inlet
Outlet: Back Detector
Outlet pressure: ambient

BACK DETECTOR (μECD)
Temperature: 300 'C (On)
Mode: Constant makeup flow
Makeup flow: 60.0 mL/min (On)
Makeup Gas Type: Nitrogen
Electrometer: On

SIGNAL 2
Data rate: 20 Hz
Type: back detector
Save Data: On
Start Save Time: 3.00 min
Stop Save Time: 30.00 min
Zero: 0.0 (Off)
Range: 0
Fast Peaks: Off
Attenuation: 0

COLUMN COMP 2
Derive from back detector

POST RUN
Post Time: 0.00 min

TIME TABLE

Time	Specifier	Parameter & Setpoint

7673 Injector

Front Injector:

Sample Washes	1	
Sample Pumps	5	
Injection Volume	1.0	microliters
Syringe Size	10.0	microliters
PostInj Solvent A Washes	2	
PostInj Solvent B Washes	2	
Viscosity Delay	0	seconds
Plunger Speed	Fast	
PreInjection Dwell	0.00	minutes
PostInjection Dwell	0.00	minutes

Back Injector:

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LABORATORY
MASTER
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Sample Washes	1
Sample Pumps	5
Injection Volume	1.0 microliters
Syringe Size	10.0 microliters
PostInj Solvent A Washes	2
PostInj Solvent B Washes	2
Viscosity Delay	0 seconds
Plunger Speed	Fast
PreInjection Dwell	0.00 minutes
PostInjection Dwell	0.00 minutes

END OF INSTRUMENT CONTROL PARAMETERS

DATA ANALYSIS PARAMETERS

Method Name: C:\MSDCHEM\1\METHODS\8081_6BF.M

Percent Report Settings

Sort By: Signal

Output Destination
    Screen: No
    Printer: Yes
    File: No

Integration Events: AutoIntegrate

Generate Report During Run Method: Yes

Signal Correlation Window: 0.020

Quantitative Report Settings

Report Type: Summary

Output Destination
    Screen: Yes
    Printer: Yes
    File: No

Generate Report During Run Method: Yes

MASTER

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Method: Q:\SVOA\GC3_GE\METHODS\8081DQ.MTH Present GC-3 method.

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Method Information

pesticide method 8081

Run Time Checklist

Pre-Run Program:	none
Name:	
Parameter:	pes
Data Acquisition:	On
Use Barcode Labels:	Off
Data Analysis:	On
Sig. 2 Mth:	none
Post-Run Program:	none
Name:	
Parameter:	

Injector Information

Injection Source:	Auto
Injection Location:	Front
Front:	
Sample Washes:	2
Sample Pumps:	2
Sample Volume:	2 stops
Viscosity Delay:	2 sec.
Solvent A Washes:	3
Solvent B Washes:	3
On-Column:	No

Purge A/B:

	Init Value	On Time (Min.)	Off Time (Min.)
A (Valve 3)	Off	1.00	0.00
B (Valve 4)	On	0.00	0.00

A - Splitless Injection:	No
B - Splitless Injection:	No

Temperature Information

Zone Temperatures:

	Set point
Inl. A	200 C.
Inl. B	Off
Det. A	300 C.
Det. B	300 C.
Aux.	Off

Oven Parameters:

Oven Equib. Time:	0.20 Min.
Oven Max:	320 C.
Oven	On
Cryo	Off

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Method: Q:\SVOA\GC3_GE\METHODS\8081DQ.MTH
Ambient: 25 C.
Cryo Blast Off

Oven Program:

	Set Point
Initial Temp:	120 C.
Initial Time:	1.00 Min.

Level	Rate (C./Min.)	Final Temp. (C.)	Final Time. (Min.)
1	12.0	300	9.00
2(A)	0.00		
3(B)			

Next Run Time: 25.00 Min.

Signal Information

Save Data: Both

Signal 1
Source: Det. A
Peak Width: 0.053 Min.
Data Rate: 5.000 Hz.
Start Data: 4.00 Min.
Stop Data: 25.00 Min.

Signal 2
Source: Det. B
Peak Width: 0.053 Min.
Data Rate: 5.000 Hz.
Start Data: 4.00 Min.
Stop Data: 25.00 Min.

Valves/Relays Information

Initial Setpoints:
5890 Valves:

Valve 1:	Off
Valve 2:	Off
Valve 3 (Purge A):	Off
Valve 4 (Purge B):	On

Detector Information

Detector	Type	State
A	ECD	On
B	ECD	On

Timed Events:

Events:	Value:	Time:
Signal Switch	OFF	0.00
Signal Switch	ON	0.00

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Inlet A Pressure Program Information

Constant Flow: Off

Pressure Program:

	Setpoint
Initial Pres.:	20.0 psi
Initial Time:	1.00 min.

Level	Rate (psi/min.)	Final Pres.(psi)	Final Time (min)
1	99.00	2.6	40.00
2(A)	0.00		
3(B)			

Total Program Time: 41.18

GC Pressure Units: psi

Fixed Values:

Column Length (m):	30.00
Column Diameter (mm):	0.530
Gas:	He
Vacuum Comp:	Off

Packed Column Information:

Equation: ---

Calibration Parameters:

	Pres. (psi)	Flow (ml/min)
1	---	---
2	---	---
3	---	---

Last pressure calibration: ---

Packed Column Flow Setting (ml/min): 0.0

Sequence Recalibration Table

Cal. Line	Cal. Level	Update Response Factor	Update Retention Times	Recalib Interval

Signal Plot Information

Signal	Attn. (2^)	Offset (%)	Time (Min.)
1	0	10	10
2	0	10	10

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Integration Events

Events:	Value:	Time:
Initial Area Reject	1	INITIAL
Initial Peak Width	0.040	INITIAL
Shoulder Detection	OFF	INITIAL
Initial Threshold	0	INITIAL

Report Specification

Destination: Report to Screen
Based on: Area
Calculations: ESTD
Printer Output: None
Report Header: None

Graphics Options

Title: Vertical
Include:
Axes Units: On
Peak Names: On
Retention Times: On
Baselines: On
Tick Marks: On
Peak Labels Font: Default 12

Calibration Settings

Title:
GC3

Reference window: 0.500 minutes
Non-reference window: 0.500 minutes
Units of amount: ng/ul
Multiplier: 1.0
RF uncal peaks: 1.0
ISTD# to adjust uncal peaks: 0
Sample Amount: 0.0

Sample ISTD Information

No Sample ISTD Amounts

Multilevel Information

Fit: Linear
Origin: Force

Calibration Table Empty

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ATTACHMENT E

Quality Control Requirements and Performance Standards for the ***Analysis of Chlorinated Pesticides by Gas Chromatography (GC)*** in Support of Response Actions under the Massachusetts Contingency Plan (MCP)

Table V B-1: Specific QC Requirements and Performance Standards for Chlorinated Pesticides (SW-846 8081B) Using WSC-CAM-V B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[2]	Required Corrective Action	Required Analytical Response Action
Initial Demonstration of Proficiency	Laboratory Analytical Accuracy & Precision	(1) Must be performed prior to using method on samples. (2) Must be performed for each matrix. (3) Must contain all target analytes. (4) Must follow procedure in Section 8.4 of SW-846 8000B.	No	NA	Refer to Section 8.4 of SW-846 8000B and Section 1.1.2 of this protocol.	NA
Retention Time Windows	Laboratory Analytical Accuracy	(1) Prior to initial calibration and when a new GC column is installed. (2) Calculated according to the method (Section 7.6 of SW-846 8000B).	No	NA	NA	NA
Endrin/DDT Breakdown	Laboratory Analytical Accuracy	(1) Before samples are analyzed and at the beginning of each 12-hour shift. (2) % Breakdown must be $\leq$15 and must be evaluated using peak areas.	Yes	(1) If DDT breakdown >20%, reject nondetect results for 4,4'-DDT. (2) If endrin breakdown >20%, reject nondetect results for endrin.	Perform injection port maintenance. Re-calibrate, if required.	Report exceedances (% breakdown >15%) and associated samples in laboratory narrative.
Initial Calibration	Laboratory Analytical Accuracy	(1) Must be analyzed at least once prior to analyzing samples, when initial calibration verification or continuing calibration does not meet the performance standards, and when major instrument maintenance is performed. (2) Minimum of 5 standards (or 6 if non-linear regression used). (3) Low standard must be $\leq$RL. (4) %RSD $\leq$20, r $\geq$0.99 (linear regression), or r^2 $\geq$0.99 (non-linear regression) for each single-component pesticide. (5) If %RSD >20, linear or non-linear regression must be used. (6) Must contain all single-component	No	NA	(1) Recalibrate as required by method. (2) If recalculated concentrations from the lowest calibration standard are outside of 70-130% recovery range, either: * The RL limit must be reported as an estimated value[3], or * The RL must be raised to the concentration of the next highest calibration standard that exhibits acceptable recoveries	Sample analysis cannot proceed without a valid initial calibration. Report non-conforming compounds (%RSD >20, r <0.99, or r^2 <0.99) in laboratory narrative. If non-linear regression (i.e., quadratic equation) is used for calibration, this must be noted in the laboratory narrative along with the compounds affected.

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Table V B-1: Specific QC Requirements and Performance Standards for Chlorinated Pesticides (SW-846 8081B) Using WSC-CAM-V B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[2]	Required Corrective Action	Required Analytical Response Action
		pesticides. (7) Multi-component analytes: Analysis of a single standard at expected mid-point of calibration range. (8) Calibration must be performed under the same conditions as the samples. (9) If linear or non-linear regression used, verify the RL by recalculating concentrations in lowest calibration standard using the final calibration curve; recoveries must be 70-130%.			when recalculated using the final calibration curve.	
Initial Calibration Verification	Laboratory Analytical Accuracy	(1) Immediately after each initial calibration. (2) Concentration level near midpoint of curve. (3) Prepared using standard source different than used for initial calibration. (4) Must contain all single-component pesticides. (5) Percent recoveries must be between 80-120% for each target analyte.	No	NA	Locate source of problem; recalibrate if >10% of all analytes are outside of criteria.	If recovery is outside of 80-120% for any analyte, report non-conforming compounds in laboratory narrative.
Continuing Calibration	Laboratory Analytical Accuracy	(1) Prior to samples, every 12 hours or every 20 samples, whichever is more frequent, and at the end of the analytical sequence. (NOTE: if internal standard calibration used, the continuing calibration at the end of the analytical sequence is not required). (2) Concentration level must alternate between low and high concentration standards (equivalent to second and fourth levels in calibration curve). (3) Must contain all single-component pesticides.	No	NA	(1) Perform instrument maintenance, reanalyze continuing calibration and/or recalibrate as required by method. (2) Renalyze "associated samples" if beginning or ending continuing calibration exhibited low response. (3) Reanalyze "associated samples" if beginning or ending continuing	Report non-conforming compounds (%D >20) and associated samples in laboratory narrative.

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Table V B-1: Specific QC Requirements and Performance Standards for Chlorinated Pesticides (SW-846 8081B) Using WSC-CAM-V B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[2]	Required Corrective Action	Required Analytical Response Action
		(4) Multi-component analytes must be verified with a one-point standard within 12 hours of being detected in a sample. (5) %D must be ≤20 for each target analyte. (6) Verify that all analytes fall within retention time windows. (7) Area count of internal standard in continuing calibration must be within ±50% of the average area count in the associated initial calibration.			calibration exhibited high response and associated pesticides were detected in the "associated samples." NOTE: "Associated samples" refers to all samples analyzed since the last acceptable continuing calibration.	
Method Blank	Laboratory Method Sensitivity (contamination evaluation)	(1) Extracted with every batch or every 20 samples, whichever is more frequent. (2) Matrix-specific (e.g., water, soil). (3) Target analytes must be <RL.	Yes	NA	(1) If concentration of contaminant in sample is <10x concentration in blank, locate source of contamination; correct problem; re-extract and re-analyze method blank and associated samples. (2) No corrective action required if concentration of contaminant in sample is >10x concentration in blank or if contaminant not detected in sample.	(1) If sample re-extraction is not possible, report non-conformance in laboratory narrative. (2) If contamination of method blanks is suspected or present, the laboratory, using a "B" or some other convention, should qualify the sample results. Blank contamination should also be documented in the laboratory narrative. (3) If re-extraction is performed within holding time and yields acceptable method blank results, the laboratory may report results of the re-extraction only. (4) If re-extraction is performed outside of holding time, the

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Table V B-1: Specific QC Requirements and Performance Standards for Chlorinated Pesticides (SW-846 8081B) Using WSC-CAM-V B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[2]	Required Corrective Action	Required Analytical Response Action
						laboratory must report results of both the initial extraction and re-extraction.
Laboratory Control Sample (LCS)	Laboratory Analytical Accuracy	(1) Extracted with every batch or every 20 samples, whichever is more frequent. (2) Concentration level near midpoint of curve. (3) Must contain all single-component pesticides.[1] (4) Matrix-specific (e.g., soil, water). (5) Percent recoveries must be between 40-140% for target analytes. (6) Must be prepared in a water-miscible solvent (e.g., acetone, methanol).	Yes	Recovery <10%; affects nondetect results for affected analyte in all samples extracted with this LCS.	(1) Locate source of problem; re-extract and re-analyze LCS and associated samples if >10% of all analytes are outside of criteria. (2) If ≤10% of compounds are outside of the acceptance criteria, re-extraction is not required as long as recoveries are >10%. (3) If >10% of compounds are above the acceptance criteria (>140%), reextraction is not required if affected compounds were not detected in associated samples.	(1) If sample re-extraction is not possible, report non-conformance in laboratory narrative. (2) If recovery is outside of 40-140% for any analyte, report non-conforming compounds in laboratory narrative. (3) If re-extraction is performed within holding time and yields acceptable LCS results, the laboratory may report results of the re-extraction only. (4) If re-extraction is performed outside of holding time and yields acceptable LCS results, the laboratory must report results of both the initial extraction and re-extraction.
LCS Duplicate	Laboratory Analytical Accuracy & Precision	(1) Extracted with every batch or every 20 samples, whichever is more frequent. (2) Concentration level near midpoint of curve. (3) Must contain all single-component pesticides.[1]	Yes	Recovery <10%; affects nondetect results for affected analyte in all samples extracted with this LCS.	(1) Locate source of problem; re-extract and re-analyze LCS and associated samples if >10% of all analytes are outside of recovery acceptance criteria.	(1) If sample re-extraction is not possible, report non-conformance in laboratory narrative. 2) If recovery is outside of 40-140% for any

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Table V B-1: Specific QC Requirements and Performance Standards for Chlorinated Pesticides (SW-846 8081B) Using WSC-CAM-V B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[2]	Required Corrective Action	Required Analytical Response Action
		(4) Matrix-specific (e.g., soil, water). (5) Percent recoveries must be between 40-140% for target analytes. (6) RPDs must be ≤20 for waters and ≤30 for solids. (7) Must be prepared in a water-miscible solvent (e.g., acetone, methanol).			(2) If ≤10% of compounds are outside of the recovery acceptance criteria, re-extraction is not required as long as recoveries are >10%. (3) If >10% of compounds are above the recovery acceptance criteria (>140%), reextraction is not required if affected compounds were not detected in associated samples.	analyte or if RPD is outside of criteria, report non-conforming compounds in laboratory narrative. (3) If re-extraction is performed within holding time and yields acceptable LCS results, the laboratory may report results of the re-extraction only. (4) If re-extraction is performed outside of holding time and yields acceptable LCS results, the laboratory must report results of both the initial extraction and re-extraction.
MS/MSD	Method Accuracy & Precision in Sample Matrix	(1) Every 20 samples (at discretion of laboratory or at request of data user). (2) Matrix-specific. (3) Concentration level near midpoint of curve. (4) Must contain all single-component pesticides.[1] (5) Percent recoveries between 30 – 150%. (6) RPDs ≤20 for waters and ≤30 for solids. (7) Must be prepared in a water-miscible solvent (e.g., acetone, methanol).	Yes ONLY when requested by the data user	Recovery <10%; affects nondetect result for affected analyte in unspiked sample only.	Check LCS; if recoveries are acceptable in LCS, narrate non-conformance.	Note exceedances in laboratory narrative.

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Table V B-1: Specific QC Requirements and Performance Standards for Chlorinated Pesticides (SW-846 8081B) Using WSC-CAM-V B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[2]	Required Corrective Action	Required Analytical Response Action
Surrogates	Method Accuracy in Sample Matrix	(1) Minimum of 2 surrogates, one that elutes at beginning of GC run and one that elutes at end of GC run. Recommended surrogates: TCMX and DCB (2) Percent recoveries must be between 30-150% for both surrogates on both columns.	Yes (report surrogate recoveries from both columns)	Recovery <10%; affects all nondetect results in affected sample.	If the same surrogate is outside of limits on both columns: (1) Re-extract the sample if surrogate recoveries are low and there is no chromatographic interference. (2) Re-extract the sample if surrogate recoveries are high and pesticides were detected in the sample. NOTES: (a) If surrogate recoveries are high and target analytes are not detected in sample, re-extraction is not required. (b) If chromatographic interference is present and surrogate recovery would cause rejection of data (i.e., < 10%), reanalyze sample on dilution. (c) If a surrogate is diluted to a concentration below that of the lowest calibration standard, reextraction and/or reanalysis is not required.	(1) Report recoveries outside of 30-150% in laboratory narrative. (2) If re-extraction yields similar surrogate non-conformances, the laboratory must report results of both the initial extraction and re-extraction. (3) If re-extraction is performed within holding time and yields acceptable surrogate recoveries, the laboratory may report results of the re-extraction only. (4) If re-extraction is performed outside of the holding time and yields acceptable surrogate recoveries, the laboratory must report results of both the initial extraction and re-extraction. (5) If sample is not re-extracted due to chromatographic interference, the laboratory must provide the chromatogram in the data report.
Internal Standards (optional)	Laboratory Analytical Accuracy and Method Accuracy in	(1) Minimum of 1. Recommended internal standard: DCB (2) Area counts in samples must be between 50 – 200% of the area counts	No	Recovery <20%; affects all nondetect results quantitated using	If internal standard is outside of limits, reanalyze sample unless chromatographic	(1) Report nonconformances in laboratory narrative. Include actual recovery

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Table V B-1: Specific QC Requirements and Performance Standards for Chlorinated Pesticides (SW-846 8081B) Using WSC-CAM-V B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[2]	Required Corrective Action	Required Analytical Response Action
	Sample Matrix	in the associated continuing calibration standard. (3) Retention times of internal standards must be within ±30 seconds of retention times in associated continuing calibration standard.		affected internal standard in associated sample.	interference present. NOTE: If chromatographic interference is present and internal standard area would cause rejection of data (i.e., <20%), reanalyze sample on dilution.	of internal standard and provide summary of analytes quantitated using the internal standard. (2) If reanalysis yields similar internal standard non-conformances, the laboratory must report results of both analyses. (3) If reanalysis is performed within holding time and yields acceptable internal standard recoveries, the laboratory may report results of the reanalysis only. (4) If reanalysis is performed outside of the holding time and yields acceptable internal standard recoveries, the laboratory must report results of both analyses. (5) If sample is not reanalyzed due to chromatographic interference, the laboratory must provide the chromatogram in the data report.
Identification and Quantitation	NA	(1) Peak area is the expected default to be used for quantitation of pesticides under most circumstances. Regardless if peak area or peak height is used, the same method used for quantitation of samples must also be used for	NA	If RPD >100 for single-component pesticides, reject positive result for affected pesticide.	If the RPD between the dual column results is >100 for single-component pesticides or >500 for multi-component pesticides,	If the RPD between the dual column results exceeds 40, the laboratory must qualify the sample results and/or note the

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Table V B-1: Specific QC Requirements and Performance Standards for Chlorinated Pesticides (SW-846 8081B) Using WSC-CAM-V B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[2]	Required Corrective Action	Required Analytical Response Action
		calibration standards. (2) The laboratory must use the average calibration factor, response factor, linear or non-linear regression curve generated from the associated initial calibration for quantitation of each single-component pesticide. (3) Secondary column analysis: Laboratory must utilize a second dissimilar column to confirm positive results. The laboratory must report the higher of the two results. All required QC parameters (e.g., calibrations, LCSs, etc.) must be met on the secondary column as well. (4) Results must be reported with 2 or more "significant figures" if ≥RL. If reporting values below the RL, report with 1 or more "significant figures".[4]		If RPD >500 for multi-component pesticide, reject positive result for affected pesticide.	reanalyze the sample on dilution. Both analyses must be reported. Alternatively, additional sample cleanup techniques may be warranted.	exceedance in the laboratory narrative. If the RPD exceedance is due to interference, the lower of the dual column values can be reported; this must be noted in the laboratory narrative.
General Reporting Issues	NA	(1) The laboratory must only report values ≥ the sample-specific reporting limit. (2) Dilutions: If diluted and undiluted analyses are performed, the laboratory should report results for the lowest dilution within the valid calibration range for each analyte. The associated QC (e.g., method blanks, surrogates, etc.) for each analysis must be reported. **NOTE: Laboratories shall not perform dilutions on samples due to sulfur interference. Laboratories must employ a cleanup technique to reduce the presence of sulfur interference.** (3) Results for soils/sediments must be reported on a dry-weight basis for comparison to MCP regulatory	NA	NA	NA	(1) Complete analytical documentation for diluted and undiluted analyses must be made available for review during an audit. (2) The performance of dilutions must be documented in the laboratory narrative or on the report form. Unless due to elevated concentrations of target compounds, reasons for dilutions must be explained in the laboratory narrative. (3) If samples are not

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Table V B-1: Specific QC Requirements and Performance Standards for Chlorinated Pesticides (SW-846 8081B) Using WSC-CAM-V B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[2]	Required Corrective Action	Required Analytical Response Action
		standards. (4) Refer to Appendix V B-1 for chain-of-custody requirements regarding preservation, cooler temperature, and holding times.				preserved properly or are not received with an acceptable cooler temperature, note the non-conformances in the laboratory narrative. (4) If samples are extracted and/or analyzed outside of the holding time, note the non-conformances in the laboratory narrative.

[1]Refer to Section 1.6 for guidance regarding the inclusion of multi-component pesticides in LCSs and MS/MSDs.
[2]As per Appendix IV of MassDEP Policy #WSC-07-350, *MCP Representativeness Evaluations and Data Usability Assessments*, September 2007, if these results are observed, data users should consider nondetect results as unusable and positive results as estimated with a significant low bias.
[3]If the RL is estimated due to unacceptable recovery of the lowest standard, the CAM RL has not been achieved; Question G of the "MassDEP MCP Analytical Protocol Certification Form" must be answered "NO" and this must be addressed in the laboratory narrative.
[4]Reporting protocol for "significant figures" is a policy decision included for standardization and consistency for reporting of results and is not a definition of "significant" in the scientific or mathematical sense.

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ESS Laboratory
Division of Thielsch Engineering
Cranston, RI

SOP NO. 60_8082

ORGANOCHLORINE PCB's AS AROCLORS BY GAS CHROMATOGRAPHY: CAPILLARY COLUMN TECHNIQUE (EPA Method 608/SW-846 METHOD 8082)

REVIEWED BY:

Operation Manager — Date 4/27/07

QA Manager — Date 4/27/07

Laboratory Director — Date 5/1/07

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ORGANOCHLORINE PCB's AS AROCLORS BY GAS CHROMATOGRAPHY: CAPILLARY COLUMN TECHNIQUE (EPA Method 608/SW-846 METHOD 8082)

1.0 SCOPE AND APPLICATION

1.1 This procedure is used to determine the concentrations of various organochlorine polychlorinated biphenyls (PCBs) as Aroclors, in extracts from solid, aqueous and waste matrices. Open-tubular, capillary columns were employed with electron capture detectors (ECD).

1.2 Although performance data are presented for many of the compound chemicals, it is unlikely that all of them could be determined in a single analysis. This limitation results because the chemical and chromatographic behavior of many of these chemicals can result in co-elution. Several cleanup/fractionation schemes are provided in this procedure. Any compound is a potential method interference when it is not a target analyte.

1.3 Several multi-component mixtures (i.e., Aroclors) are listed as target compounds. When samples contain more that one multi-component analyte, a higher level of analyst expertise is required to attain acceptable levels of qualitative and quantitative analysis. The same is true of multi-component analytes that have been subjected to environmental degradation or degradation by treatment technologies. These result in "weathered" Aroclors (or any other multi-component mixtures) that may have significant differences in peak patterns than those of standards. In these cases, individual congener analyses may be preferred over total mixture analyses.

1.4 Compound identification (single component compounds) based on single column analysis should be confirmed on a second column, or should be supported by at least one other qualitative technique. This procedure describes analytical conditions for a second gas chromatographic column that can be used to confirm the measurements made with the primary column. GC/MS is also recommended as a confirmation technique if sensitivity permits.

1.5 This procedure has a reporting limit of 0.5 ug/L for aqueous samples and 50 ug/Kg for solid and waste samples. Lower detection limits are achievable by further concentration of sample extract upon request. Mass CAM required reporting limits are 0.25ug/L for aqueous samples (achieved with a 1ml final volume) and 100 ug/Kg soil (Assuming 100% solids).

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2.0 METHOD SUMMARY

2.1 A measured volume or weight of sample (approximately 1 L for liquids, 2 g to 30 g for solids) is extracted using the appropriate sample extraction technique. Liquid samples are extracted at neutral pH with methylene chloride using a separatory

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funnel (SOP 50_3510). Solid samples are extracted with hexane-acetone (1:1) or methylene chloride-acetone (1:1) using either automate Soxhlet extraction (SOP 50_3541A) or Microwave Extraction method (SOP 50_3546). A variety of cleanup steps may be applied to the extract, depending on (1) the nature of the co-extracted matrix interferences and (2) the target analytes. After cleanup, the extract is analyzed by injecting a 1-ul sample into a gas chromatograph with a narrow- or wide-bore fused silica capillary column and electron capture detector (GC/ECD).

3.0 HEALTH AND SAFETY

3.1 Each employee has been trained and has acknowledged being trained in the safe use and handling of chemicals being used in the laboratory. This training has been performed according to the ESS Training SOP 80_0016 and by the Chemical Hygiene Plan, SOP No. 90_0001, in conjunction with the Safety orientation.

3.2 All sample and material handling should be done in a hood while using proper protective equipment to minimize exposure to liquid or vapor. Minimum personnel protective equipment includes the use of laboratory safety glasses, a lab coat or apron, and protective gloves.

3.3 The MSDs for the concentrated chemicals used in the area are kept on file in a central location that is available for the employees for review.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

4.1 Aqueous samples are collected in 1 Liter borosilicate glass jars with Teflon lined caps. The samples are stored in a dark walk-in cooler at 4° C. Two liters should be provided so samples can be re-extracted when necessary. ***Aqueous samples must be extracted within 7 days from date sampled.***

4.2 Soil / sediment samples are collected in 4 - 8 ounce jars with Teflon lined caps. The samples are stored in a dark walk-in cooler at 4° C. Thirty grams of sample is required for extraction and ten grams is required to determine the percent solids. One hundred grams should be provided so samples can be re-extracted when necessary. ***Soil / Sediment samples must be extracted within 14 days of date sampled.***

4.3 All extracts are stored in 2ml Teflon capped vials in the extract storage refrigerator located in the SVOA lab. These extracts are stored at 4°C and must be analyzed within 40 days of date extracted.

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5.0 INTERFERENCES AND POTENTIAL PROBLEMS

5.1 Sources of interference in this method can be grouped into three broad categories: (1) contaminated solvents, reagents or sample processing hardware; (2) contaminated GC carrier gas, parts, column surfaces or detector surfaces; and (3) the

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presence of co-eluting compounds in the sample matrix to which the ECD will respond. Interferences co-extracted from the samples will vary considerably from waste to waste. While general cleanup techniques are referenced or provided as part of this method, unique samples may require additional cleanup approaches to achieve desired degrees of discrimination and quantitation.

5.2 Interferences by phthalate esters introduced during sample preparation can pose a major problem in PCBs determinations. Common flexible plastics contain varying amounts of phthalate esters that are easily extracted or leached from such materials during laboratory operations. Cross-contamination of clean glassware routinely occurs when plastics are handled. Avoiding contact with any plastic materials and checking all solvents and reagents for phthalate contamination can best minimize interferences from phthalate esters. Exhaustive cleanup of solvents, reagents and glassware may be required to eliminate background phthalate ester contamination.

5.3 Glassware must be scrupulously cleaned as soon as possible after use by rinsing with the last solvent used. Follow by detergent washing with hot water and rinses with tap water. Rinse with methanol and drain. Store dry glassware in a clean environment.

5.4 Cleanup by acid partitioning (SOPs 50_3665A/3665M) is suggested for PCB samples that are extremely contaminated with particulate and oxidizable organics.

5.5 It may be difficult to quantitate Aroclor patterns and single component PCBs together. Guidance on the identification of PCBs is given in Section 8.0.

6.0 EQUIPMENT/APPARATUS

6.1 **Gas chromatograph**: an analytical system complete with gas chromatograph suitable for on-column and split/split-less injection and all required accessories including syringes, analytical columns, gases, electron capture detectors (ECD) and DOS based PC system interfaced to the GC with HP Chemstation ND EnviroQuant software.

6.1.1 **Wide-bore Columns**:

6.1.1.1 **Column 1**: RTX-CL Pesticide column I. 30 m x 0.32 mm ID (or 0.53 mm ID, if needed; GCs -3, -6, -7) fused silica capillary column.

6.1.1.2 **Column 2**: RTX-CL Pesticide II column. 30 m x 0.32 mm ID (or 0.53 mm ID, if needed; GCs -3, -6, -7) fused silica capillary column.

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6.1.2 **Miscellaneous Instrument Parts**:

6.1.2.1 Single Goose-neck glass liners from either HP or Restek.
6.1.2.2 Gold Seals and Washers (HP 05971-27305)

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6.1.2.3 Graphite Ferrules (0.53mm.)
6.1.2.4 O-rings (HP #5180-4182)
6.1.2.5 HP ceramic tiles for cutting columns.

6.2 **10 ul** Glass bore Injector **Syringe**.

6.3 **Volumetric flasks** – Class A.

6.4 **Class A pipettes** – 5 ml, 8 ml, 25 ml.

6.5 **Sample vials**: glass with Teflon-lined crimp tops, 2.0ml.

6.6 **Syringes** -25 ul, 100 ul, 500 ul, and 1000 ul .

6.7 Teflon-lined crimp tops, 2.0ml

6.8 **Data system**:

6.8.1 **Computers:** The SVOA laboratory has five GC/ECD systems capable of analyzing method 608/8082. SVOA GC's 6 and 7 have a DELL computers. SVOA GC1, GC3, and GC5 have AST computers. SVOA GC1, GC3, and GC5 have Windows 95 operating system. SVOA GC6 has the Windows 2000 operating system. SVOA GC7 has a Windows XP operating system. All computer systems are networked to a Windows 2003 server, which is the destination of all files. A differential back-up is performed nightly and a full backup is performed each weekend using Symantec Backup Exec to tapes. As the systems acquires and stores data onto the server, the server becomes full. The data is downloaded and archived on an external hard drive

6.9 **Software:** HP/Agilent Environmental Chemstation - The software is interfaced to an electron capture detector and allows the continuous acquisition and storage on machine-readable media of chromatograms obtained throughout the instrument program. The software integrates the abundance in any EICP between specified times. Current versions SVOA ECD5:G1045A 01.00, SVOA ECD1/3:G1045A C.01.00 and SVOA ECD6:G1701DA D.00.01.27.

7.0 REAGENTS AND STANDARDS

7.1 **Reagents**: All reagents should be reagent grade or pesticide grade for this analysis. Unless otherwise indicated it is intended that all reagents shall conform to specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

WORKSHEET

7.2 **Solvents**:

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7.2.1 N-hexane
7.2.2 Methylene Chloride
7.2.3 Acetone

7.3 **Standards**: Store the standard solutions (stock, composite, calibration and surrogate) at 4° C in Teflon-sealed containers in the dark, with the exception of the primary surrogate, which is stored at room temperature to prevent crystallization. In the latter case, it has been determined that the rate of surrogate standards usage is such that no loss of analytes is experienced.

7.3.1 **Primary Standards**: Expiration dates of unopened/opened primary standards are as stated in SOP 50.0006 or as stated by the manufacturer,, whichever is earlier. Copies of the certificates of analysis are on file. These certificates detail the compounds in each of the mixes. Certificates are kept on file in the laboratory. The following primary standard solutions are purchased as certified solutions:

Primary Standard	Manufacturer	Catalog #	Conc. (ug/ml)	Secondary Standard	Manufacturer	Catalog #	Conc. (ug/ml)
Aroclor 1016	Ultra	EPA-1282	1000	Aroclor 1016	Supelco	DP4 - 8097	1000
Aroclor 1221	Ultra	EPA-1292	1000	Aroclor 1221	Supelco	DP4 - 8098	1000
Aroclor 1232	Ultra	PP-301	100	Aroclor 1232	Supelco	DP4 - 4805	1000
Aroclor 1242	Ultra	PP-311	100	Aroclor 1242	Supelco	DP4 -4806	1000
Aroclor 1248	Ultra	PP-341	100	Aroclor 1248	Supelco	DP4 - 4807	1000
Aroclor 1254	Ultra	PP-351	100	Aroclor 1254	Supelco	DP4 - 4808	1000
Aroclor 1260	Ultra	EPA-1362	1000	Aroclor 1260	Supelco	DP4 -4809	1000
Aroclor 1262	Ultra	EPA-1372	1000	Aroclor 1262	Accustandard	C-262S-H-10X	1000
Aroclor 1268	Ultra	EPA-1382	1000	Aroclor 1268	Accustandard	C-268S-H-10X	1000
TMX/DCB Surr	Accustandard	CLP-032-R	200				

7.3.2 **Stock standards** are used to prepare multi-component standards. They are stored in 40 ml glass vials with Teflon lined caps for a maximum of one year from the date of preparation.

7.3.2.1 **Stock 1660 (1016/1260)**: 500 µl of each primary standard (Aroclor 1016 and 1260 from Ultra) and 100 µl of the surrogate mixture, is added to a 10 ml volumetric flask and diluted to the 10 ml mark with hexane.

7.3.2.2 **Second Source Stock 1660 (1016/1260)**: 50 µl of each primary standard (Aroclor 1016 and 1260 from Supelco) and 10 µl of the surrogate mixture, is added to a 50 ml volumetric flask and diluted to the 50 ml mark with hexane (Final Conc 1016/1260 is 50,000µg/L & Surrogate is 2000 µg/L).

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7.3.2.3 **Stock 1221, 1232, 1242, 1248, 1254, 1262, and 1268**: Each stock is prepared separately as follows: 0.5 ml of the primary standard (for 1221, 1262 and 1268 use 100µl) and 10 µl of the surrogate mixture is added to a 50 ml volumetric flask and diluted to the 50 ml mark with hexane (Final Conc Arochlors is 1000µg/L & Surrogate is 40 µg/L).

7.3.2.4 **Second Source Stock 1221, 1232, 1242, 1248, 1254, 1262, and 1268**: Each stock standard is prepared separately as follows: 50 uL of the secondary standard and 10 ul of the surrogate mixture is added to a 50 ml volumetric flask and diluted to the 50 ml mark with hexane. (Final Conc Arochlors is 1000µg/L & Surrogate is 40 µg/L).

7.3.2.5 **Surrogate Stock TCX/DCB**: 0.5 ml of primary standard (TCX/DCB Pest. /PCB Surrogate from Accustandard), is added to a 200 ml volumetric flask and diluted to the 200 ml mark with acetone. Final concentration of 0.5 ug/ml. Used as the surrogate spiking solution.

7.3.3 **Aroclor Working standards:** Store standards in 15 ml glass vials (2 ml target vials when 1 ml prepared) with Teflon lined caps. Standards expiration is six months. Concentrations should correspond to the expected range of concentrations found in real samples and bracket the linear range of the detector. They are used for the initial calibration procedure.

7.3.3.1 **Aroclor 1016 and 1260 (1660) Initial Calibration Standards**: The concentrations should correspond to the expected range of concentrations found in real samples and bracket the linear range of the detector. They are used for the initial calibration procedure. A set of 6 levels is prepared in volumetric glassware and diluted to mark with hexane. Working standards are prepared from the stock 1660 standard (7.3.2.1).

Level	Standard used	Initial Conc.	Amount added	Final volume	Final Conc.
1	stock 1660	Aroclor 50,000ug/L Surr. 2000 ug/L	25 ul	25.0 ml	Aroclor 50ug/L Surr. 2 ug/L
2	Stock 1660	Aroclor 50,000ug/L Surr. 2000 ug/L	50 ul	25.0 ml	Aroclor 100ug/L Surr. 4 ug/L
3	Stock 1660	Aroclor 50,000ug/L Surr. 2000 ug/L	250 ul	25.0 ml	Aroclor 500ug/L Surr. 20 ug/L
4	Stock 1660	Aroclor 50,000ug/L Surr. 2000 ug/L	1000 ul	50.0 ml	Aroclor 1000ug/L Surr. 40 ug/L
5	Stock 1660	Aroclor 50,000ug/L Surr. 2000 ug/L	800 ul	25.0 ml	Aroclor 1600ug/L Surr. 64 ug/L
6	Stock 1660	Aroclor 50,000ug/L Surr. 2000 ug/L	1000 ul	25.0 ml	Aroclor 2000ug/L Surr. 80 ug/L

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7.3.4 **ICV/BS/BSD/MS/MSD Solution**: The spike solutions are prepared in Acetone as follows.

Analyte	Vendor	Initial Conc.	Amount Added	Final Volume	Final Conc.
Aroclor 1016 Aroclor 1260 or project specific Aroclor	Supelco	1000 ug/ml	2 ml each	200 ml	10 ug/ml

7.3.4.1 Surrogate solution (7.3.2.3) is added to each sample, method blank, blank spike, blank spike duplicate, matrix spike and matrix spike duplicate with a 1 ml calibrated syringe. See the appropriate extraction method for volumes.

7.3.4.2 The PCB ICV/BS/BSD/MS/MSD spike solution (7.3.4) is added to each blank spike, blank spike duplicate, matrix spike, and matrix spike duplicate with a 1 ml calibrated syringe. Unless otherwise requested by the client, ESS Laboratory uses the Aroclor 1660 spike mix. See the appropriate extraction method for volumes.

8.0 PROCEDURE

8.1 Extraction:

8.1.1 In general, water samples are extracted at a neutral pH with methylene chloride as a solvent using a separatory funnel (SOP 50_3510). Extract solid samples with methylene chloride-acetone (1:1) using the automated Soxhlet extraction (50_3541A) or ultrasonic extraction (SOP 50_3550) procedures. Oil samples are diluted with Hexane using the waste dilution extraction procedure (50_3580).

NOTE: 1:1 Hexane/acetone (replacing 1:1 methylene chloride-acetone) may be more effective as an extraction solvent for organochlorine PCBs in some environmental and waste matrices than is methylene chloride/acetone. Use of hexane/acetone generally reduces the amount of co-extracted interferences and improves signal/noise.

8.1.2 Spiked samples are used to verify the applicability of the chosen extraction technique to each new sample type. Each sample type must be spiked with the compounds of interest to determine the percent recovery and the limit of detection for that sample. See section 10.0 for guidance on demonstration of initial method proficiency as well as guidance on matrix spikes for routine sample analysis.

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8.2 Cleanup/Fractionation:

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8.2.1 Cleanup procedures may not be necessary for a relatively clean sample matrix, but most extracts from environmental and waste samples will require additional preparation before analysis. The specific cleanup procedure used will depend on the nature of the sample to be analyzed and the data quality objectives for the measurements.

8.2.1.1 When PCBs are to be measured in a sample, Acid Partitioning (SOPs 50_3665A/3665M) is recommended. This is followed with Sulfur Cleanup by Copper (SOP 50_3660) or GPC (SOP No. 50_3640A).

8.3 **Instrument Set Up**:

8.3.1 Typical operating conditions are as follows:

Inlet	EPC	Split/Splitless
Carrier gas	Helium	Hydrogen
Make-up gas	Nitrogen	Nitrogen
Head Pressure	6-7 psi	9 psi
Total Flow	70-100 ml/min	70-100 ml/min
Column Flow (0.32 column)	2-3 ml/min	2-3 ml/min
Detector Makeup Pressure	45-50 psi	45-50 psi
Detector Makeup Flow	50-60 ml/min	50-60 ml/min
Temperature program:		
Injector temperature	250°C	250°C
Detector temperature	320°C	320°C
Initial temperature/Time	130°C for 1 min	130°C for 1 min
Ramp	18°C/min	18°C/min
Final temperature/Time	310°C for 4 min	310°C for 4 min

Conditions may change; e.g. GC-5 uses Methane/Argon as carrier gas.

8.4 **Initial Calibration**:

NOTE: When PCBs are to be quantitatively determined as Aroclors, the initial calibration consists of two parts:

1) A standard containing a mixture of Aroclors 1016/1260 (1660) will include many of the peaks represented in the other seven Aroclor mixtures. Consequently, such a standard is used to demonstrate the linearity of the detector and that a sample does not contain peaks that represent any one of the Aroclors. This standard is also used to determine the concentrations of either Aroclor 1016 or 1260 when

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present in a sample. Therefore, an initial 6-point calibration is performed using the mixture of Aroclors 1660.

2) Standards of the other seven Aroclors are necessary for pattern recognition. These standards are also used to establish a single-point calibration factor for each Aroclor, after the Aroclor 1660 mixture has been used to describe the detector response. The standards for these seven Aroclors should be analyzed before the analysis of any samples, and may be analyzed before or after the analysis of the six 1660 standards.

8.4.1 ESS Laboratory's policy is that the audit trail on the Chemstation/Enviroquant software is always on. This ensures that any changes made to the instrument operating method be documented through the audit trail.

8.4.2 All acceptance criteria for initial and continuing calibration apply to both the primary and secondary columns.

8.4.3 **Priming the Column**: Once the chromatographic system operating conditions have been established, calibration may begin. Because of the low concentration of PCB standards injected on a GC/ECD, column adsorption may be a problem when the GC has not been used for a day or more. Therefore, the GC column may be primed or de-activated by injecting a PCB standard mixture approximately 20 times more concentrated than the mid concentration standard. Inject this standard mixture prior to beginning the initial calibration or calibration verification.

CAUTION: Carryover may be observed in the injection just following this system priming. Always run an acceptable blank prior to running any standards or samples.

8.4.4 **Loading the instrument**: All standards and samples are transferred with disposable pipettes into 2 ml target vials designed to fit the HP auto-sampler. The target vials are labeled with the ID of the standard or sample using a fine point marker. The tray on the auto-sampler is numbered 1-100. The instrument is set up with two independent injection systems, which means that the contents of one vial can not be injected into both injection ports at the same time. The 6-level standards are placed in slots 1-6. This is the standard setup for the calibration standards. Vials can be placed in different slots as long as the slot number is written in the logbook.

8.4.5 **Log Book**: All samples set up on the instrument must be entered into the run logbook. All logbook entries are done prior to sample analysis. The logbook must be filled out completely with the date, vial number (slot number), computer file number, method number, ESS lab ID, and the initials of the analyst setting up the instrument.

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8.4.5.1 Date includes the day, month, and year.

8.4.5.2 Vial number: This field has to be filled in for each entry.

8.4.5.3 Computer file ID is an abbreviation of the path and file associated with a particular vial number. (e.g. vial #3 analyzed on 3/12/05 has a computer file ID of 031205003)

8.4.5.4 The ESS Lab ID includes the ID of the standards, samples and all QC samples.

8.4.5.5 The Initials are signed by the analyst setting up the instrument.

8.4.5.6 The Comment section is a summary of calibration results, dilution information, and any unusual observations. (Ex.: carry over information into the sample, retention time shift, calibration standard is less than 15% Recovery, dilution information, CCV injection time, etc...)

8.4.5.7 The Method section is the method in the chromatographic software used to operate the instrument. All 6-level standards are injected like the sample using the instrument set-up criteria in section 8.3.

8.4.6 **Starting a run with Chemstation Software**: The GC is controlled through Chemstation. The methods are set up with the instrument set-up information from section 8.3. All of the operating parameters are saved in Chemstation under method file ID's. The P8082 method operates the front and rear injectors. These methods are saved in the C:\HPCHEM\ directory. A copy of the Chemstation method is in **Attachment** A. To run the instrument:

8.4.6.1 Open the Chemstation icon. From the Chemstation Menu select Sequence.

8.4.6.2 In Initial Setup type the name of the directory where data will be stored. The directory file is Q:\SVOA\Instr\Data\MMYY\Date. (e.g. If samples were set up to run on 9/8/05 on GC5 then the directory would be called Q:\SVOA\GC5_gg\Data\gg0905\gg090805)

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8.4.6.3 In Analytical Sequence, type in the vial numbers that correspond to when the front and rear injectors will start and stop analyzing.

8.4.6.4 In the Sample List type in the IDs of the samples and standards next to the corresponding vial numbers.

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8.4.6.5 Select Save to save the newly created sequence. Save the sequence with the day created or the ID. (e.g.: A sequence saved on Monday is saved as Mon.s. Sequences do not have to be saved and can be easily recreated. Saving a sequence can save time if for any reason the analytical sequence needs to be restarted due to an instrument or computer malfunction.

8.4.6.6 Start the analysis by selecting RUN Analytical Sequence.

8.4.6.7 A Chemstation data file will be created for each sample and standard. These files will be stored in the directory C:\HPCHEM\1\data.

8.4.7 Reviewing the Initial Calibration Data: All GC Chemstation Data is reduced with the EnviroQuant software. (Refer to EnviroQuant Operator's Manual).

8.4.7.1 Setting up an EnviroQuant Method: All PCB methods are set up as documented in **Attachment** A. With each new calibration, the response and absolute retention times are updated. The absolute retention times are from the **Aroclor level 4 standards**.

8.4.7.2 Updating a New Initial Calibration:

8.4.7.2.1 Load the Aroclor method with parameters set according to 8.4.7.1.

8.4.7.2.2 Save the method using a unique identification such as 8082AD (the next initial calibration will be saved as 8082AE, then 8082AF, etc.). Note that the ID of this new method is written in the Method column of the RUN log next to the Initial Calibration Standard ID's)

8.4.7.2.3 Convert all Chemstation Files to EnviroQuant files.

8.4.7.2.4 Load **the Level -4** standard file for the 1660. Go into QEDIT and correctly integrate all of the peaks. Three to five peaks must be integrated for each Aroclor. Choose peaks that are at least 25% of the height of the largest Aroclor peak.Qdelete any false positives. Go into the initial calibration and update the absolute retention times and responses. Each of the Aroclors must resemble the patterns of chromatograms in **Attachment** C. If the pattern of an Aroclor does not match then refer to the following table:

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Problem	Corrective Action
Wrong standard injected	Check sequence: setup section 8.4.6. Make sure standards are correctly placed on tray.
Contaminated standard	Inject another aliquot of standard. If that doesn't work, remake standard.
Standard prepared incorrectly	Remake standard
A dirty injection port	Perform maintenance. Refer to section 18.0

8.4.7.2.6 For Standards levels 1,2,3,5,6 only update responses.

8.4.7.2.7 The software calculates the average response factor from the 6-level curve. (See calculations 9.0) The %RSD of the response factor in each of the 6-levels is calculated and must be less than 20% (must be less than 10% in Method 608). If any of the PCBs are outside of this criterion, check the data setup in the EnviroQuant software and review the integration of each of the 6-levels and make sure all responses were correctly updated. If manual integrations need to be made, update responses again.

8.4.7.2.8 When updating responses, all peaks in each level should fall within the retention time windows established according to section 8.5. If the peaks have drifted outside of the retention time windows then the initial calibration is not acceptable. This peak drift is usually due to a carrier gas leak or clog in a gas line. Most leaks come from the injection port. To correct a leak, start by tightening all connections, including the column nut, gold seal and insert retainer nut. If this doesn't work, go to section 18.0 and replace the ferrule, the septa, and the o-ring with a new one. In some cases, the gooseneck liner will get clogged with a piece of septa. Inspect the liner for pieces of septa. If the problem persists, consult with supervisor or a service representative. Once the problem has been corrected, reanalyze the entire initial calibration

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8.4.7.2.9 For those analytes of interest which are greater than 20% RSD (10% for Method 608), use a linear calibration. The linear calibration is a regression equation that does not pass through zero. See Calculations in section 9.0. The instrument response is treated as the dependent variable (y) and the concentration as the independent variable (x). The regression will produce the slope and intercept terms for a linear equation in the form:

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$$y = ax + b$$

Where:

y= instrument response (peak area)
a= Slope
x= Concentration
b= intercept

8.4.7.2.10 The regression calculation will generate a linear least squares regression ($r \geq 0.995$) or a non-linear regression ($r^2 \geq 0.99$) In the latter case, a six point minimum calibration shall be used for second order and a seven point minimum calibration shall be used for third order.

8.4.7.2.11 Once all integrations are correct, the %RSD is less than 20%, $r \geq 0.995$ or $r^2 \geq 0.99$ and all standards are within the RT windows, resave the method.

8.4.7.2.12 Evaluate quantitation at the low standard. This is the standard at the reporting limit. Typically PCB aroclors are reported to the 100ppb standard. Make a copy of the low standard file and requant the copy. Requanting the low standard should produce results within 70-130% of the true value. The calibration range, switching between an average and linear calibration, the standards, and the condition of the instrument will affect this result. If this criterium is not met, make the appropriate corrections (ie maintainence) and recalibrate.

8.4.7.2.13 Document all maintenance performed in the instrument maintenance log.

8.4.7.2.14 After the 1660 initial calibration curve has been established the analyst will analyze a 1000 ug/L second source standard to confirm the calibration. The second source must be within 20% of the known value. ***The acceptance criterion for DoD or MADEP CAM is 20%.***

8.4.7.2.15 Each of the remaining 7 Aroclor stock standards are analyzed to establish a one point calibration with the corresponding second source for each used for confirmation. These Aroclor methods will be used to quantitate samples until the next 1660 initial calibration.

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8.4.7.2.16 Use this new method to analyze samples and continuing calibration standards (calculate and generate report).

8.5 Retention Time Windows

8.5.1 Retention time windows are central to the identification of 3 to 5 peaks used to identify an Aroclor. Absolute retention times are used for compound identification in all GC and HPLC external standard methods. Retention time windows are established to compensate for minor shifts in absolute retention times as a result of sample loadings and normal chromatographic variability. The width of the retention time window should be carefully established to minimize the occurrence of both false positive and false negative results.

8.5.2 Determining Retention Time Windows:

8.5.2.1 Before establishing retention time windows, make sure that the chromatographic system is operating reliably and that the system conditions are optimized for the target analytes and surrogates in the sample matrix to be analyzed.

8.5.2.2 Make three injections of the mid-range CCV Aroclor over the course of a 72-hour period. Serial injections or injections over a period of less than 72 hours may result in retention time windows that are too tight.

8.5.2.3 Record the retention time in minutes for each peak used for quantitation and surrogate to three decimal places. Calculate the mean and standard deviation of the three absolute retention times for three to five peaks used for calibration or identification and each surrogate.

8.5.2.4 If the standard deviation of the retention times for any peak used for calibration is 0.000 (i.e., no difference between the absolute retention times), then use a default standard deviation of 0.01 minutes. The width of the retention time window for three to five peaks used for calibration or identification and surrogates is defined as ± 3 times the standard deviation of the mean absolute retention time established during the 72-hour period or 0.03 minutes, whichever is greater.

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8.5.2.5 Establish the center of the retention time window for each Aroclor peak and surrogate by using the absolute retention time for each peak and surrogate from the mid-range CCV (Level 4 standard) at the beginning of each analytical shift. For samples run during the same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration.

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8.5.2.6 Absolute retention time windows must be calculated for each Aroclor peak and surrogate on each chromatographic column and instrument used.

8.5.2.7 New retention time windows must be established when a new GC column is installed or when GC column has been shortened during maintenance.

8.5.2.8 Typically all PCB extracts are acid washed prior to analysis. In cases where an acid wash is not performed, a DDT/DDD/DDE standard must be analyzed to ensure that no aroclor peaks interfere with these analytes. If aroclor peak coelute with DDT,DDE or DDE adjust GC conditions to obtain better resolution.

8.6 Surrogates are added to each sample, blank, QC sample and calibration standard. Surrogate retention times in the calibration standards are useful in tracking retention time shifts. Whenever the observed retention time of a surrogate is outside of the established retention time window for standards, the analyst is advised to determine the cause and correct the problem before continuing analyses.

8.7 **Sample Analysis**: Once the initial calibration has passed all of the quality control criteria. Sample analysis may begin. All samples and standards must be run under the same conditions as the initial calibration.

8.7.1 **Sequence of Analysis:** Prior to sample analysis calibration verification (CCV) standards are analyzed. The CCV is the AR1660 Level 4 standard or, in the case of Navy/DoD samples, two standards from the initial calibration at or below the middle of the calibration curve alternated throughout the analytical sequence. ESS Laboratory analyzes four most common Aroclors, (**AR1242, AR1248, AR1254, and AR1660**) at the level 4 standard concentration. AR1660 is used to verify the initial calibration. The other Aroclors are analyzed for pattern recognition. Samples are immediately analyzed after the standards. Samples include the method blank, blank spike, blank spike duplicate, samples, matrix spikes, and matrix spike duplicates. Samples cannot be analyzed more than 12 hours after the first standard is injected and no more than 20 samples (10 field samples for DoD) can be analyzed within that 12 hour period. The calibration must also be verified at the end of each analytical sequence. The continuing calibration verification consists of the same four stock standards. The continuing calibration verification at the end of each analytical sequence consists of the Aroclor 1660 stock to verify detector response. If samples contain AR1221, AR1232, AR1262 or AR1268, samples must be re-analyzed and bracketed with the appropriate standards.

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NOTE: Analysis of a second source calibration verification standard is performed immediately after initial calibration in place of the CCV.

NOTE: It is not acceptable practice to group QC samples together and/or to analyze QC samples on one instrument and their associated samples on another instrument. Analyst must try to analyze batch QC samples, as capacity allows, along with their associated field samples.

8.7.2 **Loading calibration standards and samples**: See section 8.4.3.

8.7.3 **Log Book entries**: See section 8.4.4.

8.7.4 **Running Continuing Calibration Standards and Samples with Chemstation Software:** See section 8.4.5

8.7.5 **Reviewing Continuing Calibration Standard and Sample data:** The AR1660 CCV is quantitated using the average response factor from the 6-point calibration. The calibration factor for each peak *should* not exceed a ±20 percent difference/drift when compared to the initial calibration curve. The average %D of 3-5 peaks from each Aroclor ***must*** not exceed ±20 %D (20% for DOD/CAM). For the remaining Aroclors a single point (mid-level) standard is analyzed. All GC Chemstation data is reduced with the Enviroquant Software. NOTE: %Difference is used when average response factors are used in the initial calibration and %Drift is used for linear regression. (Refer to Hewlett Packard's EnviroQuant Operators Manual)

8.7.5.1 Update the AR1660 Method with the initial AR1660 Continuing Calibration standards:

8.7.5.1.1 Open the Enviroquant Icon on the computer connected to the instrument that ran the samples and standards.

8.7.5.1.2 Load the method with the initial calibration data that corresponds to the time and place where the samples and standards ran. See section 8.5.6. Usually this is the last method entered into the logbook.

8.7.5.1.3 Convert all Chemstation files to Enviroquant Files

8.7.5.1.4 Load the appropriate Aroclor standard that ran just prior to the first sample set up in the analytical sequence. Go into QEDIT and correctly integrate all of the peaks. Qdelete any false positives. Go into the initial calibration and update the absolute retention times only. **Do not update responses!** If the pattern of the Aroclor does not

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match the corresponding pattern in **Attachment** C then refer to the table in section 8.5.6.2.4.

8.7.5.1.5 Save the updated method according to section 8.5.6.2.2.

8.7.5.1.6 Perform the same procedure (8.7.5.1.1-8.7.5.1.5) for the other Aroclors as needed (only update retention times).

8.7.5.2 Reviewing the Continuing Calibration Standards:

8.7.5.2.1 Use the method updated in section 8.7.5.1.5 to analyze (calculate and generate report) the AR1660 calibration standards which ran before and after the samples of interest.

8.7.5.3 If the retention times have shifted, see section 8.4.6.2.7. All of the retention times must also be within the established Retention Time windows (see section 8.5.6.1.1). When these criteria are exceeded, inspect the gas chromatographic system to determine the cause and perform whatever maintenance is necessary before verifying calibration and proceeding with sample analysis. (See section 8.3) If routine maintenance does not return the instrument performance to meet the QC requirements based on the last initial calibration, then a new initial calibration must be performed.

8.7.6 Sample injection may continue for as long as the calibration verification standards and standards interspersed with the samples meet the QC requirements. The sequence ends when the standards which follow a set of samples have been injected or when qualitative and/or quantitative QC criteria are exceeded.

8.7.6.1 Each sample analysis must be bracketed with an acceptable initial calibration and ICV or calibration verification standard(s) (each 12 hour shift), followed by another set of calibration standards. All samples that were injected after the standard that last met the QC criteria must be re-injected.

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8.8 Sample Data Reduction:

8.8.1 All samples bracketed within acceptable AR1660 calibration standards are analyzed by the same method used to analyze the standards. If Aroclors other than 1016 or 1260 are present, single point calibration standards analyzed just prior to the samples. The initial calibration generated with the current 1660 initial calibration is used for quantitation. Through EnviroQuant, a quantitation report and chromatogram is generated for each sample.

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8.8.2 The EnviroQuant software will tentatively identify Aroclor 1016 or 1260 only when a peak from a sample extract falls within the daily retention time windows.

8.8.3 The analyst must carefully review the chromatograms to ensure that all peaks were identified correctly. If there is interference in the sample and the chromatograms are not very clean, the analyst must go into Qedit section of the software to review the integrations.

8.8.3.1 Each blank, blank spike, blank spike duplicate, sample, matrix spike, and matrix spike duplicate should have surrogates identified. If the surrogate has not been identified, go into Qedit and integrate the peak in the surrogate retention time window. In some cases, surrogates can not be identified due to interferences from unknown components that elute at the same time as the surrogate. This must be noted in the case narrative. If the surrogates are outside the retention time window, then the retention times have shifted. This is either due to a gas leak, a clog in the gas line, or the sample matrix. When this happens, the sample must be re-analyzed. Oily samples can cause retention time shifts. The analyst may need to dilute sample to aid in identification (reduce matrix affect) but to meet reporting limit of 1 ppm, the sample result must be quantitated on the undiluted sample.

8.8.3.2 The analyst must also review the baseline on all sample and standard chromatograms. A baseline rise will result from contamination of the injection port and column. When the baseline interferes with sample and standard analysis, samples and standards must be re-analyzed. Attempt to isolate the sample or samples that contaminated the system. These samples should be cleaned with florisil or re-analyzed with a dilution.

8.8.3.3 Late eluting compounds can carryover from sample to sample or sample to standard. Large unresolved peaks in a sample are a good indication of carryover. If carryover occurs, first identify the sample, which is potentially carrying over. Rerun samples with carryover contamination. Run instrument blanks, consisting of clean hexane, after the sample that carried over until carryover is eliminated. In cases when carryover is too great, the sample may need to be cleaned up further or diluted.

8.8.4 The on-column concentration of each Aroclor peak for Aroclor 1016 and 1260 in the sample is provided on the quantitation report along with the surrogate concentrations. The concentration is in ng/ml. If there are no peaks

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that resemble an Aroclor according to the sample chromatogram then Aroclors are not present in the sample

8.8.5 If there is a group of peaks that resemble an Aroclor in **Attachment** C then that Aroclor standard must be analyzed before and after the sample. To quantitate an Aroclor, first make sure all of the major peaks for the specific Aroclor are present. All major peaks are specified in **Attachment** C. Next compare the pattern of the peaks in the sample to the corresponding standard which was analyzed just prior. The ratio of area counts of two major peaks in the sample should be equal to the ratios of the same peaks in the standard. In some cases the ratios will not match due to interferences from other components in the sample. Identifying interferences is the analyst's judgement. If the sample is mixed in with a difficult matrix with much interference then more than one analyst should attempt to identify the PCB. Once the analyst is convinced that the PCB is present, the Aroclor can be quantitated; otherwise, report as ND and narrate. When the match is unclear and reported as ND, the potential presence of PCB must be narrated. In this case chromatograms should be provided. Use three-five peaks for quantitation. The peaks with the least amount of interference should be used for quantitation. The ratio between the three-five peaks should also come close to matching the standard. The software is set up to quantitate each peak individually. The ratio is comparable when the quantitation of each peak generates the same value. If the average Aroclor concentration is below the calibration range then it cannot be reported as detected.

8.8.6 If the average Aroclor concentration is above the MDL and below the level 6 concentration, then the result is tentative and must be confirmed.

8.8.7 If the average Aroclor concentration is above the level 6 concentration, then the sample needs to be diluted to bring the concentration of the analyte to within the level 1 and level 6 ranges.

8.8.8 A PCB Aroclor is confirmed by performing the same analysis on a different column. If the sample was initially analyzed on an RTX-CL Pesticide column I and there was an analyte detected above the method detection limit, the sample must be confirmed on an RTX-CL Pesticide Column II. The reverse also holds true.

8.8.9 The final result is determined by comparing the initial analysis with the confirmation analysis.

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8.8.9.1 If, on the confirmation analysis, the sample is at a concentration less than the method detection limit (MDL), then the result is reported as not detected (ND) above the method reporting limit (MRL). The MRL is based on the level 1 calibration standard concentration. **NOTE: When samples are analyzed for the DoD , results above**

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the MDL that are not confirmed on the second column are Q-flagged.
Discussion to be included in project narrative.

8.8.9.2 If the confirmation result is within calibration range, the result has been confirmed. The initial result is compared to the confirmation result and if the RPD< 40%, then the higher result is reported. If the RPD > 40%, the analyst must evaluate the chromatogram for co-eluting peaks. If no co-elution is detected and all other QC is within criteria, report the higher result. If co-elution was present, the analyst is to report the result without co-elution and an explanation is to be added to the case narrative.

8.8.9.3 If the confirmation results are at a concentration greater than the level 6 standard, then a dilution must be made. Dilutions should be 60-80% of the highest standard. The sample dilution is analyzed. The criterion stated in 8.7.9.2 is applied to the two results.

NOTE: ESS Laboratory normally uses level 2 to determine the MRL of soil samples but reserves the option of utilizing the level 1 standard.

9.0 CALCULATIONS

9.1 Calculate PCB soils as follows:

$$\text{PCB Result ug/Kg} = \frac{\text{(Avg Concentration of 3-5 Peaks ppb)(Extract Volume ml)(Dilution)}}{\text{(\% Solid/100)(Weight of Sample Measured g)}}$$

9.2 Calculate PCB aqueous as follows:

$$\text{PCB Result ug/L} = \frac{\text{(Avg Concentration of 3-5 Peaks ppb)(Extract Volume ml)(Dilution)}}{\text{Volume of Sample in ml}}$$

9.3 Calculate PCB matrix spike recoveries as follows:

9.3.1 Matrix spike added is calculated as follows:

$$\text{Matrix Spike Added} = \frac{\text{(Conc. of Spike)(Volume of Spike Added)}}{\text{Initial Volume}}$$

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NOTE: Initial volume can be in liters or grams. If it is a soil make sure to multiply initial volume by % solids/100.

9.3.2 Matrix spike % recovery is calculated as follows:

$$\text{\% Recovery} = \text{Matrix Spike Result X 100}$$

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Matrix Spike Added

9.4 For surrogate recoveries, % RSD and all other calculations done by the computer's software refer to Hewlett Packard © 1992 printed in USA 11/92 Part No. HP G1032-90020.

9.5 Calibration Factor (CF)

$$CF = \text{Peak Area} / \text{Mass of Compound Injected}$$

9.6 Percent Relative Standard Deviation:

$$\%\ RSD = (SD / \text{Average CF}) \times 100\%$$

Where: SD = standard deviation (o-1)

9.7 Percent Difference: $\%\ D = ((CFv - CF\ ave) / CF\ ave) \times 100\%$

Where:
CFv = Calibration Factor of the verification standard.
CF ave = The mean calibration factor from the initial calibration.

9.8 Percent Drift:

$$\%\ Drift = \frac{\text{Calculated Concentration} - \text{Theoretical Concentration}}{\text{Theoretical Concentration}} \times 100\%$$

10.0 QUALITY ASSURANCE/QUALITY CONTROL

10.1 Immediately after the initial calibration, a second source standard (ICV) is analyzed. This standard is prepared at the level 4 concentration. The percent recovery between the Level 4 in the initial calibration and the second source must be 20-120%. If the percent recovery is outside criterion, then prepare and re-analyze the ICV. If the second consecutive ICV is still outside criterion, then a new initial calibration must be performed with freshly prepared calibration standards. Maintain instrument when necessary. Sample analysis cannot begin until a valid second source has been analyzed.

10.2 **Accuracy and Precision**: All laboratory personnel must demonstrate initial proficiency for each sample preparation method/matrix that he/she performs. All new employees must successfully demonstrate initial proficiency prior to independently performing analysis on real samples. This must be accomplished by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The initial proficiency results will become part of each employee's training file

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10.2.1 QC Sample Preparation: Four QC samples must be prepared from a spiking solution with the analytes of interest. The spiking solution must be made using standards **prepared independently from those used for calibration**. The samples must be prepared at a concentration that would result in data falling at the mid-level standard of the calibration curve. In most cases the blank spike solution is used. Prep: The samples are prepared in a clean matrix (DI water for aqueous samples and Ottawa sand or Sodium sulfate for soil/solids).

10.2.2 Sample Analysis: The four QC samples must be analyzed within the criteria of the method being evaluated. The QC samples must be handled in exactly the same manner as actual samples.

10.2.3 Accuracy Calculation: Accuracy is defined as the closeness of agreement between an observed value and an accepted reference value. Each of the four spiked samples will be calculated for percent recovery. The average of the percent recovery values is the accuracy result.

10.2.4 Precision Calculation: Precision is defined as the agreement of a set of replicate measurements without assumption of knowledge of the true value. Precision is estimated by the relative standard deviation (RSD) of the four QC samples.

$$\%RSD = (s / \bar{x})\ 100\ \%$$

Where:

s = Standard Deviation of a finite number of values. On a scientific calculator use the **σxn-1** key.

$\bar{x}$ = the average of the four QC sample % recoveries.

10.2.5 Reporting Accuracy and Precision: Accuracy and Precision data should be presented with the following minimum info:

Matrix: Prep Method:
Clean-up Method: Analysis Method:
Date Extracted: Date Analyzed:
Prepared by: If Applicable Analyzed by:
Accuracy: Precision:

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Parameter	*% Rec. QC 1*	*% Rec. QC 2*	*% Rec. QC 3*	*% Rec. QC 4*	*Average Recovery*	*Standard Deviation*	*%RSD*

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10.2.6 Interpretation of Results: The percent recoveries should be between 60-130% and the %RSD should be less than 30%. If any of the accuracy and precision results do not fall within the criteria then re-prep and reanalyze all QC samples only for those analytes that did not pass.

10.3 A Method Blank is extracted with each 20 samples or analytical batch. A method blank is analyzed with each analytical sequence to ensure the instrument is clean. The concentration of analytes in the method blank must be less than ½ MRL (MRL is the Level 1 standard). If the concentration of any analyte exceeds ½ MRL, then proceed to Section 11.0 for corrective action.

10.4 Run a 6-point initial calibration curve, using the primary source standards each time major instrument maintenance occurs, or if the CCV does not meet acceptance criteria. Acceptance criteria are presented in Section 8.5

10.5 A blank spike/blank spike duplicate is analyzed with each batch of 20 or less samples. The acceptance limit for the BS/BSD is 40-140% and 20%RPD for Aqueous and 30%RPD for soil/solid. ***Refer to Attachment F for MADEP CAM criteria. See Table 1 and Attachment E for DoD blank spike acceptance criteria.*** If the BS/BSD is not within limits, see section 11.0 for corrective action.

10.6 Calculate surrogate standard recovery on all samples, blanks, and spikes. Determine if the recovery is within 30-150% ***Refer to Attachment F for MADEP CAM criteria. See Table 1 and Attachment E for DoD surrogate acceptance criteria.*** If recovery is not within limits, the following are required:

10.6.1 Confirm that there are no errors in calculations, surrogate solutions and internal standards. Also, check instrument performance.

10.6.2 Examine chromatograms for interfering peaks and for integrated areas.

10.6.3 Recalculate the data and/or re-analyze the extract if any of the above checks reveal a problem.

10.6.4 If the same surrogate is outside limits on both columns, re-extract the sample. If both surrogates are outside limit on only one column, re-analyze the sample. A case narrative should be filled out in order to inform client.

10.7 Continuing calibration verification (CCV) must be performed at the beginning, after every 20 samples (10 field samples for DoD) and at the end of each analytical sequence. The response factors for the calibration should be within ±20 % difference/drift of the initial calibration (***DoD: all analytes within ± 20% of the expected value from the ICAL; refer to Attachment F for MADEP CAM criteria***). When a CCV is out of this acceptance window, re-prep and/or re-analyze the CCV. If corrective action does not produce a second consecutive (immediate) CCV within acceptance limits, then the analyst may demonstrate the initial calibration is valid by

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analyzing two consecutive CCVs at two concentrations. If CCV criteria are not acceptable, then a new initial calibration must be performed. All samples analyzed after the last acceptable CCV must be re-analyzed with the following exceptions:

10.7.1 For CCVs that are exceeded high, samples with results that are non-detect may be reported. It has been shown that results would have been detected.

10.7.2 For CCVs that are exceeded low, samples with results above a regulatory limit may be reported. **NOTE: When analyzing samples for DoD, all samples associated with a CCV below lower control limit must be re-analyzed.**

10.8 A matrix spike and matrix spike duplicate are analyzed with every batch of 20 samples. The acceptance limits 40-140% Recovery and ± 50% RPD for Aroclors. (MAMCP RPD <20% waters, <30% Soils) If the blank spike (BS) results are acceptable and the matrix spike/matrix spike duplicate results are outside of QC limits, note in case narrative. ***See Table 1 and Attachment E for DoD blank spike acceptance criteria***.

10.9 Control charts are generated quarterly for blank spikes (BS) on both Soil and Water.

10.10 MDLs are determined in reagent water and Ottawa sand/sodium sulfate and verified annually. See SOP 110_0013 for complete MDL study instructions. (Project-specific requirements may require that an MDL study be performed in the site-specific matrix.).

11.0 DATA VALIDATION

11.1 Data validation will be accomplished by reviewing all of the quality control parameters and assuring that they are within recommended ranges by completing the Data Review Checklist for GC/ECD PCB. The only exceptions made to ranges would be the following:

11.1.1 For MS/MSD, the RPD should be ± 50% (MAMCP RPD <20% waters, <30% Soils) (***Table 1 and Attachment E***). However, there are cases where duplicates may not work. If this is the case, inform client in narrative concerning sample non-homogeneity.

11.1.2 For matrix spikes, the % Recovery should be 40-140% (***Table 1 and Attachment E)***. If the matrix spike is outside criterion, check the BS/BSD. If the BS/BSDs are within limits, matrix interferences are present and should be noted in the narrative.

11.1.3 Analytical batches with Method blanks greater than or equal to the MRL ***(½ the MRL for DoD)*** will be re-prepped and re-analyzed with the following exceptions:

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11.1.3.1 Samples that are that are at least twenty times higher than the method blank may be reported.

11.1.3.2 If the analyte is found in the method blank at or above the MRL ***(½ the MRL for DoD)*** but is not in any of the associated samples, no corrective action is needed.

11.1.3.3 Any results that are reported with method blank contamination must be B-flagged and narrated.

11.1.4 For the BS/BSD, the % Recovery should be 40-140% ***(refer to Attachment F for MADEP CAM criteria;, see Table 1 and Attachment E for DoD criteria)***. If the BS/BSD is outside this criterion, the analytical batch will be re-extracted and re-analyzed with the following exceptions:

11.1.4.1 For BS/BSDs greater than the upper control limit, samples with results below the MRL may be reported. It has been shown that the results above MRL would have been detected.

11.1.4.2 In some instances there may be insufficient sample to re-extract. The client is to be contacted for instructions on how to proceed

11.1.4.3 Any samples that are reported with invalid BS/BSD data must have a notation in the case narrative.

11.2 All unusual observations and method deviations will be noted in the narrative accompanying the data report presented to the client.

11.3 A second analyst reviews all data for accuracy. Results of this review are noted on the SVOA Analysis Map in the second level review field and in the comment section.

12.0 REFERENCES

12.1 Method 8082, Test Methods for the Analysis of Solid Waste, Third Edition, Update III.

12.2 HP GC EnviroQuant User's Guide, HPG1045A.

12.3 HP Environmental Data Analysis User's Guide HPG0032C.

12.4 HP 5890 Series II /HP 6890 GC Operations Manuals.

12.5 NELAC/NELAP, Chapter 5, June 2003.

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12.6 DoD QSM, Final Version 4.1, April 2009

12.7 Massachusetts DEP WSC-CAM IIA

13.0 POLLUTION PREVENTION and WASTE MANAGEMENT

13.1 ESS Laboratory's policies on pollution prevention and waste management are covered in SOP 90_0002, Hazardous Waste Contingency and Emergency Response Plan. All employees are trained in the requirements of the SOP.

14.0 METHOD PERFORMANCE

14.1 Precision and Accuracy data must be generated by all employees before performing this analysis on client samples. The data is generated by analyzing a method blank and four blank spike samples. Acceptance criteria are 60-130% Recovery and %RSD of $\leq$ 30%.

14.2 The precision and accuracy data in Table 1 were developed using the Soxtherm extraction method. Values are in ug/L.

15.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

15.1 Typical Precision and Accuracy data generated 6/1/2005 using the Soxtherm extraction method (3541).

Compound	Spk	Avg	%RSD	%Rec
Aroclor 1016	5	4.68	5.9	93.5
Aroclor 1260	5	4.45	8.4	89.0

16.0 DEFINITIONS

16.1 **Accuracy**: The closeness of agreement between an observed value and an accepted reference value. When applied to a set of observed values, accuracy will be a combination of a random component and of a common systematic error (or bias) component.

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16.2 **Batch**: A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 20, then each group of 20 samples or less will all be handled as a separate batch.

16.3 **Bias**: The deviation due to matrix effects of the measured value (x_s - x_u) from a known spiked amount, where x_s is the spiked sample and x_u is the un-spiked sample. Bias can be assessed by comparing a measured value to an accepted reference value

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in a sample of known concentration or by determining the recovery of a known amount of contaminant spiked into a sample (matrix spike).

16.4 **Control Sample**: A QC sample introduced into a process to monitor the performance of the system.

16.5 **Equipment Blank**: A sample of analyte-free media which has been used to rinse the sampling equipment. It is collected after completion of decontamination and prior to sampling. This blank is useful in documenting adequate decontamination of sampling equipment.

16.6 **Method Reporting Limit**: The lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The MRL is generally 5 to 10 times the MDL. ESS Laboratory sets the MRL to the lowest non-zero standard in the calibration curve or higher.

16.7 **Field Duplicates**: Independent samples which are collected as close as possible to the same point in space and time. They are two separate samples taken from the same source, stored in separate containers, and analyzed independently. These duplicates are useful in documenting the precision of the sampling process.

16.8 **Blank Spike (BS)**: A known matrix spiked with compound(s) representative of the target analytes. This is used to document laboratory performance.

16.9 **Matrix**: The component or substrate (e.g., surface water, drinking water) which contains the analyte of interest.

16.10 **Matrix Duplicate**: An intralaboratory split sample which is used to document the precision of a method in a given sample matrix.

16.11 **Matrix Spike**: An aliquot of sample spiked with a known concentration of target analyte(s). The spiking occurs prior to sample preparation and analysis. A matrix spike is used to document the bias of a method in a given sample matrix.

16.12 **Matrix Spike Duplicates**: Intralaboratory split samples spiked with identical concentrations of target analyte(s). The spiking occurs prior to sample preparation and analysis. They are used to document the precision and bias of a method in a given sample matrix.

16.13 **Method Blank**: An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is carried through the complete sample preparation and analytical procedure. The method blank is used to document contamination resulting from the analytical process.

16.14 **Method Detection Limit (MDL)**: The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is

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greater than zero and is determined from analysis of a sample in a given matrix type containing the analyte. See SOP 110_0013 for further explanation.

16.15 **Organic-Free Reagent Water**: For volatiles, all references to water in the method refer to water in which an interferant is not observed at the method detection limit of the compounds of interest. A water purification system is used to generate organic-free deionized water.

16.16 **Surrogate**: An organic compound which is similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which is not normally found in environmental samples.

17.0 PERSONNEL QUALIFICATIONS

17.1 Analysts who perform this analysis must have a working knowledge or quantitative and qualitative analysis, instrumental methods of analysis, chemical laboratory methods, and equipment.

17.2 All analysts, before performing any analysis, participate in the ESS Laboratory training program (SOP80_0016). The training process consists of reading the Standard Operating Procedure, gaining instruction on the procedure from an experienced analyst, and performing the initial demonstration of capability.

18.0 TROUBLESHOOTING

18.1 **Instrument Maintenance**: The following procedure is performed when the instrument is initially set up, on a weekly basis to ensure optimum performance, or when a continuing calibration has failed the QC criteria.

18.1.1 Set the GC system to room temperature.

18.1.2 Turn off oven.

18.1.3 Remove column by unscrewing the column in the injection port.

18.1.4 Remove septum nut and septa. Discard septa.

18.1.5 Remove insert retainer nut. This will expose the O-ring and glass liner. Using a set of tweezers, remove O-ring and liner. If O-ring is not distorted then set aside for later use. Otherwise, replace O-ring. Remove the glass liner. Rinse liner with methanol and scrub with a cotton swab. If the liner is visibly stained, then replace with a new one.

18.1.6 With cotton swab dipped in methanol, clean the injection port and insert retainer nut.

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18.1.7 Remove the gold seal nut located on the bottom of the injection port. With a cotton swab and methanol, clean the gold seal.

18.1.8 Replace all parts in the following order:

18.1.8.1 Gold seal nut. Hand tighten and 1/4 turn with wrench.

18.1.8.2 Insert clean or new glass liner.

18.1.8.3 Place O-ring over liner. Slide O-ring over and down the liner until it fits snug against the injection port.

18.1.8.4 Replace insert retainer nut.

18.1.8.5 Place new green septa into insert retainer nut.

18.1.8.6 Replace septum nut. **Only hand tighten!**

18.1.8.7 Slide column nut and a new graphite ferrule over column.

18.1.8.8 Using a ceramic tile, cut 3-6 inches of the column. The cut must be square with no jagged edges.

18.1.8.9 Connect column to injection port by inserting 3 mm. of column into the injection port and hand-tighten column nut then adding 1/4 turn with a wrench.

18.1.9 Make sure all gases are flowing. (Measure flows with bubble meter.) The flow should be between 5 and 6 ml/min.

18.1.10 Turn on injection port temperature.

18.1.11 Set oven temperature to 120 ^{0}C and allow the system to stabilize. Bake out the oven at 320°C for an hour. Reset back to 120°C.

18.2 Record all maintenance in the instrument's maintenance logbook.

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19.0 DATA MANAGEMENT AND RECORDS

19.1 **Data Management** - ESS Laboratory's utilizes the Promium Element LIMS system as part of its Data Management system. Client sample information is entered into ELEMENT LIMS and analyses are assigned to each sample. The LIMS allows EPA hold times, minimum batch QC requirements, and QC criteria to be assigned to each analysis. Standards can be entered and assigned to QC samples through the LIMS. Once analysis has been performed, data is imported using DataTool avoiding

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manual errors. In conjunction with Crystal Reports, the ELEMENT system allows for a wide variety of reporting formats.

19.2 **Records** – The specific retention periods required in the NELAC Standards, EPA-CFR and state and local statutes are followed or exceeded. At a minimum, data records are retained for five years from last use (10 years for drinking water). If there is a question about whether a record should be retained or disposed because no specific requirement could be found, the record is retained until such time as a retention period is specified. Records are stored in specified-labeled locations and are easily retrievable. All raw data associated with testing is also retained including; computer printouts, chromatograms, review forms, and logbooks.

20.0 ATTACHMENTS

20.1 Table 1- DoD QSM Appendix D, QA requirements

20.2 Figure 1 – SVOA Analysis Map

20.3 Attachment A – Chemstation/Enviroquant Method

20.4 Attachment B – DELETED.

20.4 Attachment C – Chromatograms of Arochlor PCBs

20.5 Attachment E - Summary of Method Quality Objectives (DOD QSM v4.1)

20.6 Attachment F – Specific QC Requirements for WSC-CAM-V B

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Table 1 DoD Quality Systems Manual Blank Spike QC Limits

	DoD			
	Aqueous		**Soil**	
Analyte	LCL	UCL	LCL	UCL
Aroclor 1016	25	145	40	140
Aroclor 1221	-	-	-	-
Aroclor 1232	-	-	-	-
Aroclor 1242	-	-	-	-
Aroclor 1248	-	-	-	-
Aroclor 1254	-	-	-	-
Aroclor 1260	30	145	60	130
Aroclor 1262	-	-	-	-
Aroclor 1268	-	-	-	-
Decachlorobiphenyl	40	135	60	125
Tetrachloro-m-xylene	-	-	-	-

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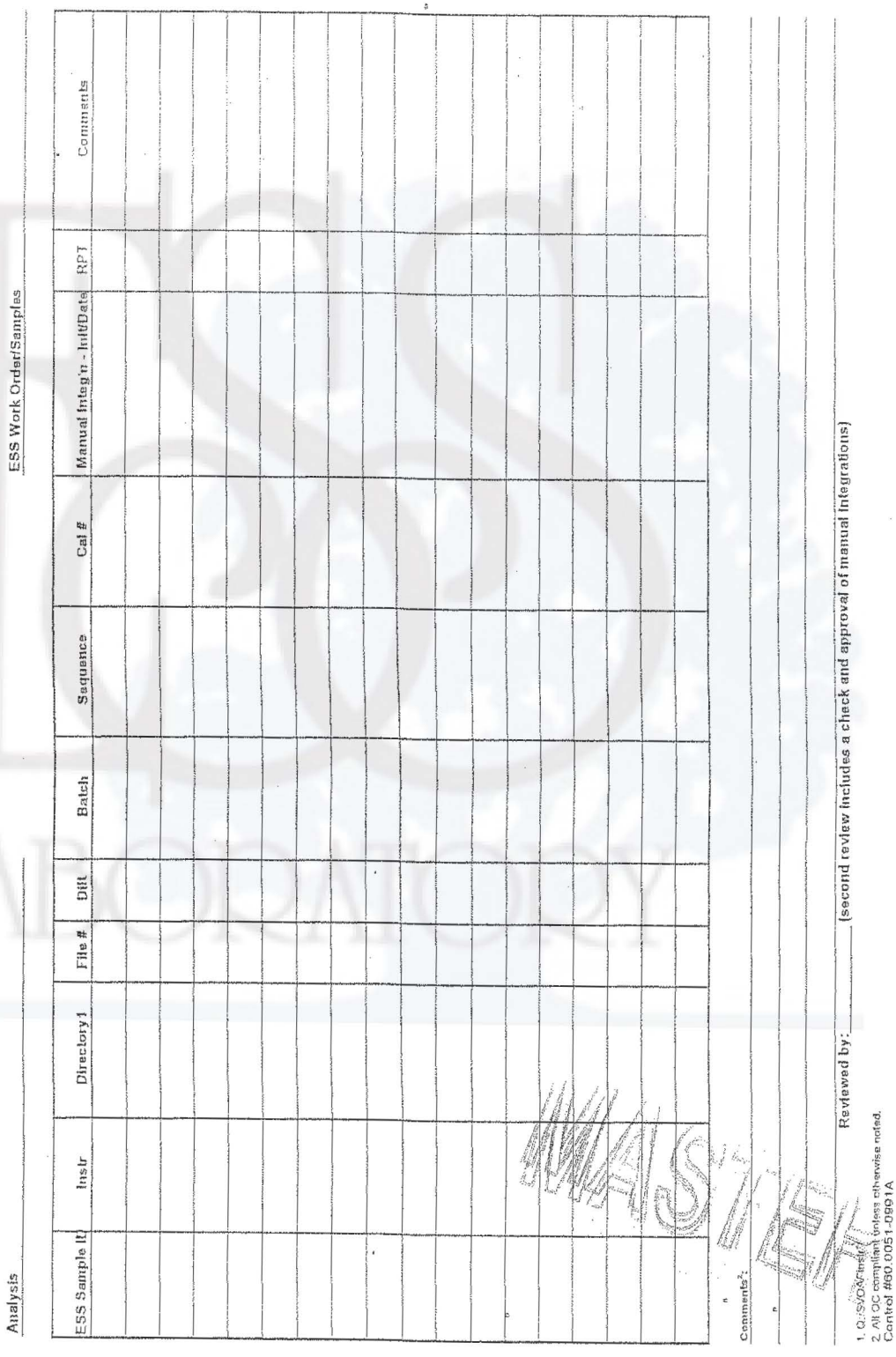
Figure 1 - SVOA Analysis Map



SVOA Analysis Map

Page ____ of ____

Analysis _____ ESS Work Order/Samples _____

ESS Sample ID	Instr	Directory1	File #	Dilt	Batch	Sequence	Cal #	Manual Integ'n - Init/Date	RPT	Comments

Comments[2]: _____

_____

_____

Reviewed by: _____ (second review includes a check and approval of manual integrations)

1. Q:\SVOA\msd\
2. All QC compliant (unless otherwise noted.
Control #60.0051-0991A

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Attachment A – Run Time Checklist
Attachment B – Certificates of Analysis

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Run Time Checklist

Pre-Run Program:	none
Name:	
Parameter:	
Data Acquisition:	On
Use Barcode Labels:	Off
Data Analysis:	On
Sig. 2 Mth:	none
Post-Run Program:	none
Name:	
Parameter:	

Injector Information

Injection Source:	Auto
Injection Location:	Front
Front:	
Sample Washes:	2
Sample Pumps:	3
Sample Volume:	2 stops
Viscosity Delay:	3 sec.
Solvent A Washes:	2
Solvent B Washes:	2
On-Column:	No

Purge A/B:

	Init Value	On Time (Min.)	Off Time (Min.)
A (Valve 3)	Off	0.50	0.00
B (Valve 4)	On	0.50	0.00

A - Splitless Injection:	No
B - Splitless Injection:	No

Temperature Information

Zone Temperatures:

	Set point
Inl. A	250 C.
Inl. B	250 C.
Det. A	320 C.
Det. B	320 C.
Aux.	Off

Oven Parameters:

Oven Equib. Time:	0.20 Min.
Oven Max:	340 C.
Oven	On
Cryo	Off
Ambient:	25 C.
Cryo Blast	Off

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Oven Program:

	Set Point
Initial Temp:	130 C.
Initial Time:	1.00 Min.

Level	Rate (C./Min.)	Final Temp. (C.)	Final Time. (Min.)
1	18.0	310	3.00
2(A)	0.00		
3(B)			

Next Run Time: 14.00 Min.

Signal Information

Save Data: Both

Signal 1

Source:	Det. A
Peak Width:	0.053 Min.
Data Rate:	5.000 Hz.
Start Data:	3.00 Min.
Stop Data:	29.25 Min.

Signal 2

Source:	Det. B
Peak Width:	0.053 Min.
Data Rate:	5.000 Hz.
Start Data:	3.00 Min.
Stop Data:	29.25 Min.

Valves/Relays Information

Initial Setpoints:

5890 Valves:

Valve 1:	Off
Valve 2:	Off
Valve 3 (Purge A):	Off
Valve 4 (Purge B):	On

Detector Information

Detector	Type	State
A	ECD	On
B	ECD	On

Timed Events:

Events:	Value:	Time:
Signal Switch	OFF	0.00
Signal Switch	ON	0.00

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Sequence Recalibration Table

Cal. Line	Cal. Level	Update Response Factor	Update Retention Times	Recalib Interval

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Signal Plot Information

Signal	Attn. (2^)	Offset (%)	Time (Min.)
1	4	10	5
2	4	10	5

Integration Events

Events:	Value:	Time:
Initial Area Reject	1	INITIAL
Initial Peak Width	0.040	INITIAL
Shoulder Detection	OFF	INITIAL
Initial Threshold	0	INITIAL

Report Specification

Destination: Report to Screen
Based on: Area
Calculations: Percent
Printer Output: None
Report Header: None

Graphics Options

Title: Vertical
Include:
- Axes Units: On
- Peak Names: Off
- Retention Times: On
- Baselines: On
- Tick Marks: On

Peak Labels Font: Default 12

Calibration Table Empty

No Calibration Settings

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Run Time Checklist

Pre-Run Program:	none
Name:	
Parameter:	
Data Acquisition:	On
Use Barcode Labels:	Off
Data Analysis:	On
Sig. 2 Mth:	none
Post-Run Program:	none
Name:	
Parameter:	

Injector Information

Injection Source:	Auto
Injection Location:	Front
Front:	
Sample Washes:	2
Sample Pumps:	3
Sample Volume:	2 stops
Viscosity Delay:	3 sec.
Solvent A Washes:	2
Solvent B Washes:	2
On-Column:	No

Purge A/B:

	Init Value	On Time (Min.)	Off Time (Min.)
A (Valve 3)	Off	0.50	0.00
B (Valve 4)	On	0.50	0.00

A - Splitless Injection:	No
B - Splitless Injection:	No

Temperature Information

Zone Temperatures:

	Set point
Inl. A	250 C.
Inl. B	200 C.
Det. A	320 C.
Det. B	320 C.
Aux.	Off

Oven Parameters:

Oven Equib. Time:	0.20 Min.
Oven Max:	360 C.
Oven	On
Cryo	Off
Ambient:	25 C.
Cryo Blast	Off

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Method: C:\HPCHEM\1\METHODS\8082BP.MTH

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Oven Program:

	Set Point
Initial Temp:	130 C.
Initial Time:	1.00 Min.

Level	Rate (C./Min.)	Final Temp. (C.)	Final Time. (Min.)
1	18.0	310	4.00
2(A)	0.00		
3(B)			

Next Run Time: 15.00 Min.

Signal Information

Save Data: Both

Signal 1

Source:	Det. A
Peak Width:	0.053 Min.
Data Rate:	5.000 Hz.
Start Data:	3.00 Min.
Stop Data:	29.25 Min.

Signal 2

Source:	Det. B
Peak Width:	0.053 Min.
Data Rate:	5.000 Hz.
Start Data:	3.00 Min.
Stop Data:	29.25 Min.

Valves/Relays Information

Initial Setpoints:
5890 Valves:

Valve 1:	Off
Valve 2:	Off
Valve 3 (Purge A):	Off
Valve 4 (Purge B):	On

Detector Information

Detector	Type	State
A	ECD	On
B	ECD	On

Timed Events:

Events:	Value:	Time:
Signal Switch	OFF	0.00
Signal Switch	ON	0.00

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Sequence Recalibration Table

Cal. Line	Cal. Level	Update Response Factor	Update Retention Times	Recalib Interval

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Signal Plot Information

Signal	Attn. (2^)	Offset (%)	Time (Min.)
1	4	10	7
2	4	10	7

Integration Events

Events:	Value:	Time:
Initial Area Reject	1	INITIAL
Initial Peak Width	0.040	INITIAL
Shoulder Detection	OFF	INITIAL
Initial Threshold	0	INITIAL

Report Specification

Destination: Report to Screen
Based on: Area
Calculations: Percent
Printer Output: None
Report Header: None

Graphics Options

Title: Vertical
Include:
- Axes Units: On
- Peak Names: Off
- Retention Times: On
- Baselines: On
- Tick Marks: On

Peak Labels Font: Default 12

Calibration Table Empty

No Calibration Settings

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Attachment B – Certificates of Analysis

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6K14001

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Certificate of Analysis

Aroclor 1260 Solution

Product Number: EPA-1362 **Page:** 1 of 1

Lot Number: R-0988Z **Lot Issue Date:** Aug-2006 **Expiration Date:** Oct-2010

This Certified Reference Material (CRM) was manufactured and verified in accordance with ULTRA's ISO 9001:2000 registered quality system, and the analyte concentrations were verified by our ISO 17025 accredited laboratory. The true value and uncertainty value at the 95% confidence level for each analyte, determined gravimetrically, is listed below.

Analyte	CAS#	Analyte Lot	True Value
Aroclor 1260	011096-82-5	E0216	1002 ± 5 µg/mL

Matrix: isooctane (2,2,4-trimethylpentane)

Balances used in the manufacture of this standard are calibrated with weights traceable to NIST in compliance with ANSI/NCSL Z-540-1 and ISO 9001.

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Edward Fitzgerald

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ISO 17025 Accredited A2LA Cert. No. 0851.01
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401-294-9400 Fax: 401-295-2330
www.ultrasci.com
Dr. Edward Fitzgerald, Senior Scientist

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6K14004

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Aroclor 1016 Solution

Product Number: EPA-1282 **Page:** 1 of 1

Lot Number: CC-2302 **Lot Issue Date:** Aug-2006 **Expiration Date:** Sep-2010

This Certified Reference Material (CRM) was manufactured and verified in accordance with ULTRA's ISO 9001:2000 registered quality system, and the analyte concentrations were verified by our ISO 17025 accredited laboratory. The true value and uncertainty value at the 95% confidence level for each analyte, determined gravimetrically, is listed below.

Analyte	CAS#	Analyte Lot	True Value
Aroclor 1016	012674-11-2	F216A	1003 ± 5 µg/mL

Matrix: isooctane (2,2,4-trimethylpentane)

Balances used in the manufacture of this standard are calibrated with weights traceable to NIST in compliance with ANSI/NCSL Z-540-1 and ISO 9001.

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Edward Fitzgerald

Dr. Edward Fitzgerald,
Senior Scientist

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9B10087-94

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Aroclor 1242 Solution

Product Number: PP-311 **Page:** 1 of 1
Lot Number: CE-3255 **Lot Issue Date:** Sep-2008 **Expiration Date:** Oct-2016

This Certified Reference Material (CRM) was manufactured and verified in accordance with ULTRA's ISO 9001:2000 registered quality system, and the analyte concentrations were verified by our ISO 17025 accredited laboratory. The true value and uncertainty value at the 95% confidence level for each analyte, determined gravimetrically, is listed below.

Analyte	CAS#	Analyte Lot	True Value
Aroclor 1242	053469-21-9	K104401	100.3 ± 0.5 µg/mL

Matrix: hexane

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William J. Leary
Quality Assurance Manager

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Aroclor 1248 Solution

Product	PP-341			**Page:**	1 of 1
Lot Number:	CE-0290	**Lot Issue Date:**	Jan-2008	**Expiration Date:**	Feb-2012

This Certified Reference Material (CRM) was manufactured and verified in accordance with ULTRA's ISO 9001:2000 registered quality system, and the analyte concentrations were verified by our ISO 17025 accredited laboratory. The true value and uncertainty value at the 95% confidence level for each analyte, determined gravimetrically, is listed below.

Analyte	CAS#	Analyte Lot	True Value
Aroclor 1248	012672-29-6	NT01718	100.3 ± 0.5 µg/mL

Matrix: hexane

MASTER

ULTRA uses balances calibrated with weights traceable to NIST in compliance with ANSI/NCSL Z-540-1 and ISO 9001, and calibrated Class A glassware in the manufacturing of these standards.

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Quality Assurance Manager

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Aroclor 1254 Solution

Product Number: PP-351 **Page:** 1 of 1

Lot Number: CB-1796A **Lot Issue Date:** Sep-2008 **Expiration Date:** Oct-2012

This Certified Reference Material (CRM) was manufactured and verified in accordance with ULTRA's ISO 9001:2000 registered quality system, and the analyte concentrations were verified by our ISO 17025 accredited laboratory. The true value and uncertainty value at the 95% confidence level for each analyte, determined gravimetrically, is listed below.

Analyte	CAS#	Analyte Lot	True Value
Aroclor 1254	011097-69-1	NTO1719	100.1 ± 0.5 µg/mL

Matrix: hexane

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Quality Assurance Manager

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5B10106, 107

Aroclor 1262 Solution

Product	EPA-1372			**Page:**	1 of 1
Lot Number:	S-0435Z	**Lot Issue Date:**	May-2007	**Expiration Date:**	Jun-2011

This Certified Reference Material (CRM) was manufactured and verified in accordance with ULTRA's ISO 9001:2000 registered quality system, and the analyte concentrations were verified by our ISO 17025 accredited laboratory. The true value and uncertainty value at the 95% confidence level for each analyte, determined gravimetrically, is listed below.

Analyte	CAS#	Analyte Lot	True Value
Aroclor 1262	037324-23-5	A-0814	1005 ± 5 µg/mL

Matrix: isooctane (2,2,4-trimethylpentane)

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Balances used in the manufacture of this standard are calibrated with weights traceable to NIST in compliance with ANSI/NCSL Z-540-1 and ISO 9001.

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Attachment C – Chromatograms of Arochlors

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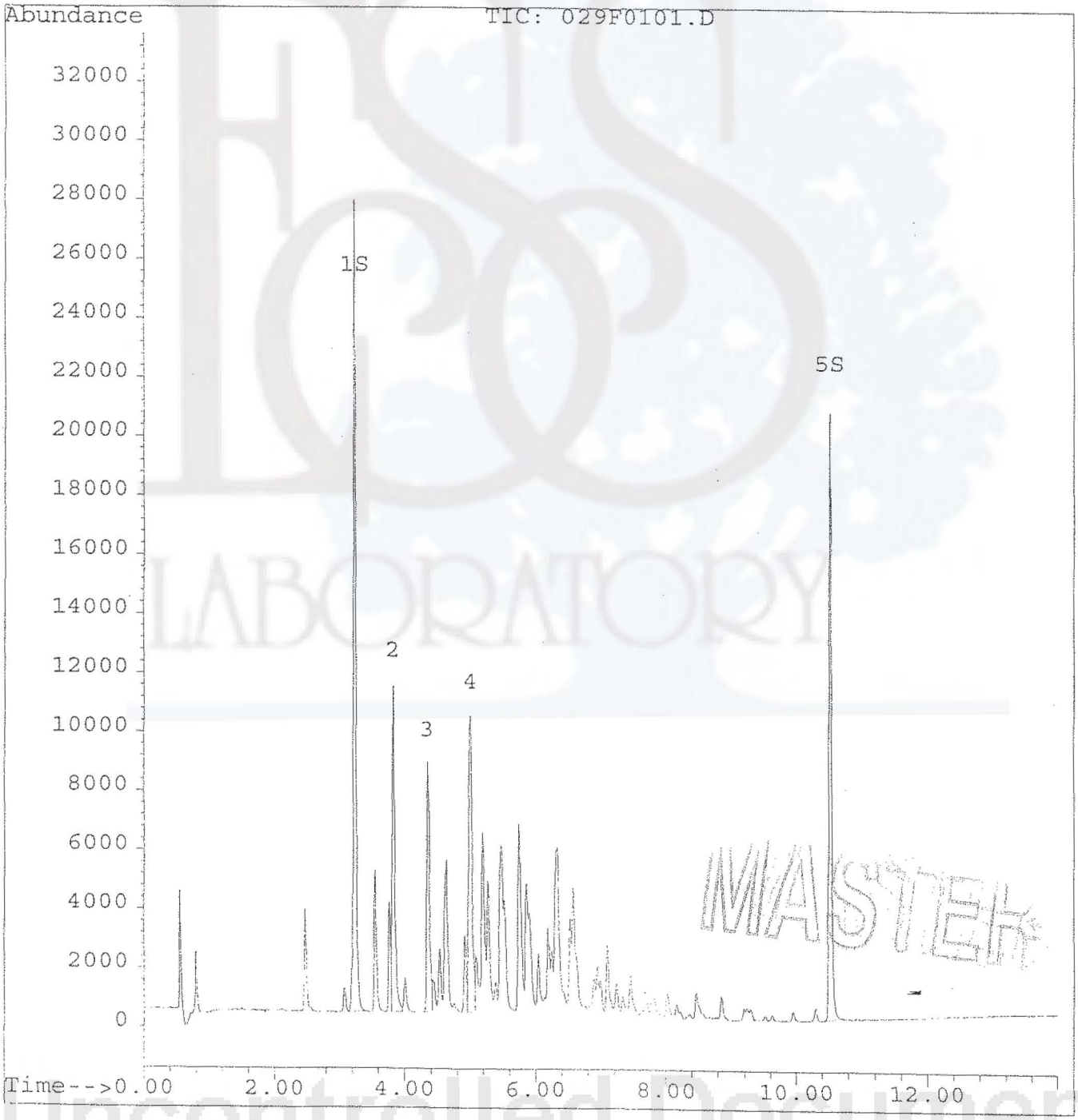
Quantitation Report

Data File : C:\HPCHEM\1\DATA\GE08209A\029F0101.D
Acq On : 21 Aug 98 08:12 AM
Sample : A32-3
Misc :
Quant Time: Aug 21 10:11 1998

Vial: 29
Operator: [GC]MS
Inst : GC 3
Multiplr: 1.00

Method : C:\HPCHEM\1\METHODS\A32F0820.M
Title :
Last Update : Fri Aug 21 10:13:49 1998
Response via : Multiple Level Calibration

Volume Inj. : 1 uL
Signal Phase : RTX-CLPESTICIDE
Signal Info : 0.53



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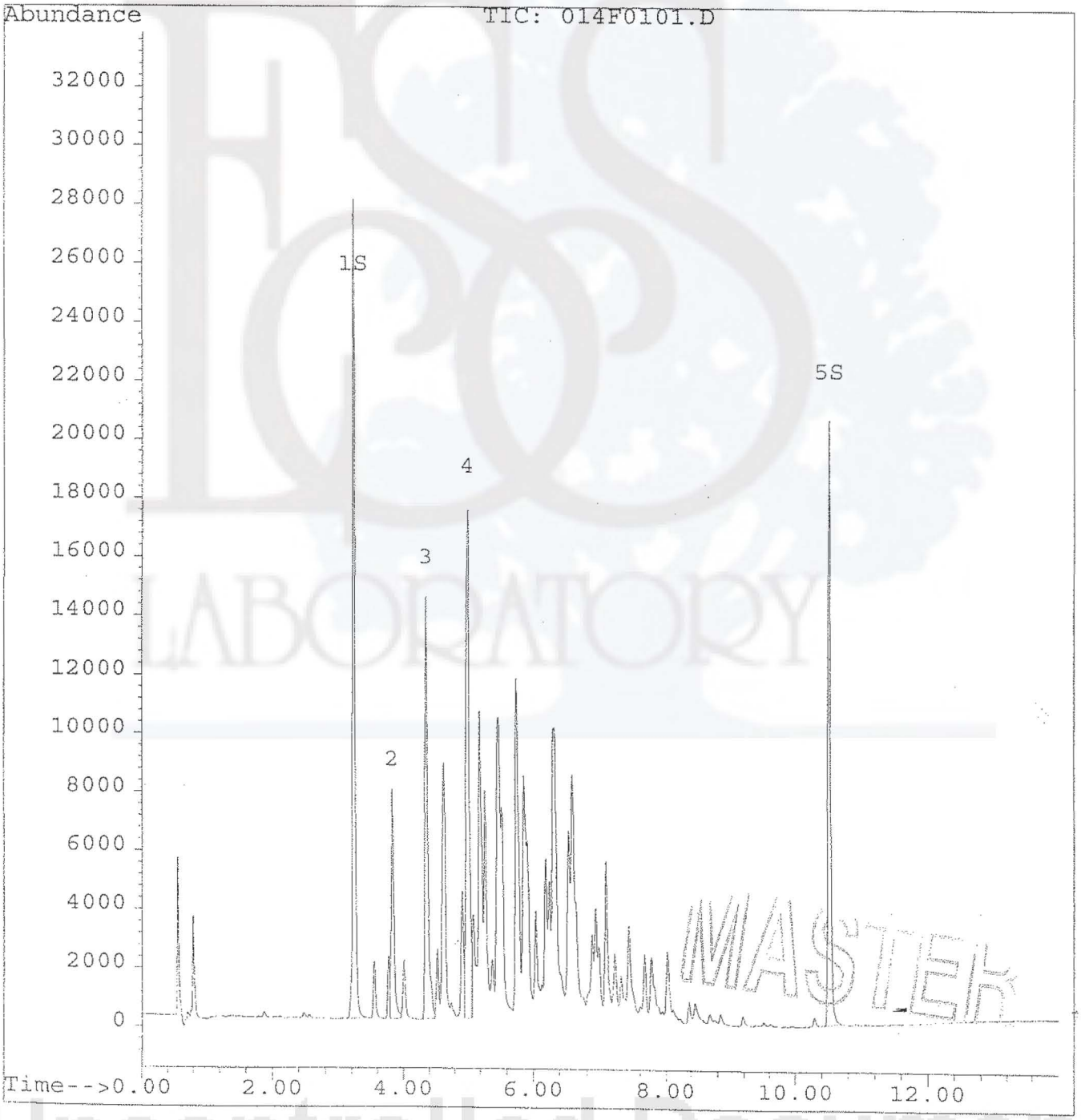
Quantitation Report

Data File : C:\HPCHEM\1\DATA\GE08209A\014F0101.D
Acq On : 20 Aug 98 07:04 PM
Sample : A42-3
Misc :
Quant Time: Aug 21 9:42 1998

Vial: 14
Operator: [GC]MS
Inst : GC 3
Multiplr: 1.00

Method : C:\HPCHEM\1\METHODS\A42F0820.M
Title :
Last Update : Fri Aug 21 09:48:46 1998
Response via : Multiple Level Calibration

Volume Inj. : 1 uL
Signal Phase : RTX-CLPESTICIDE
Signal Info : 0.53



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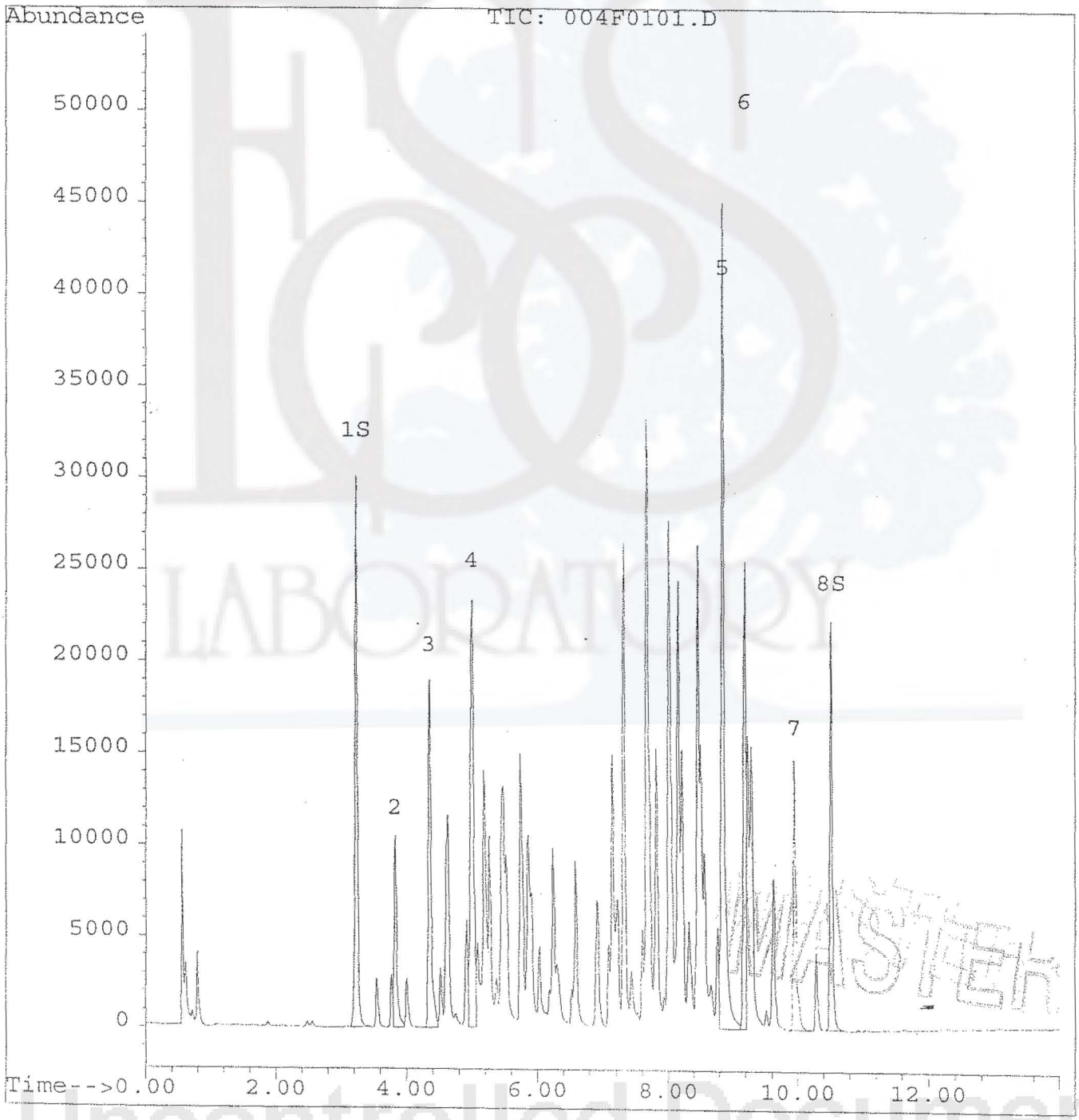
Quantitation Report

Data File : C:\HPCHEM\1\DATA\GE08209A\004F0101.D
Acq On : 20 Aug 98 04:18 PM
Sample : A60-3
Misc :
Quant Time: Aug 21 9:13 1998

Vial: 4
Operator: [GC]MS
Inst : GC 3
Multiplr: 1.00

Method : C:\HPCHEM\1\METHODS\A60F0820.M
Title :
Last Update : Fri Aug 21 09:23:30 1998
Response via : Multiple Level Calibration

Volume Inj. : 1 uL
Signal Phase : RTX-CLPESTICIDE
Signal Info : 0.53



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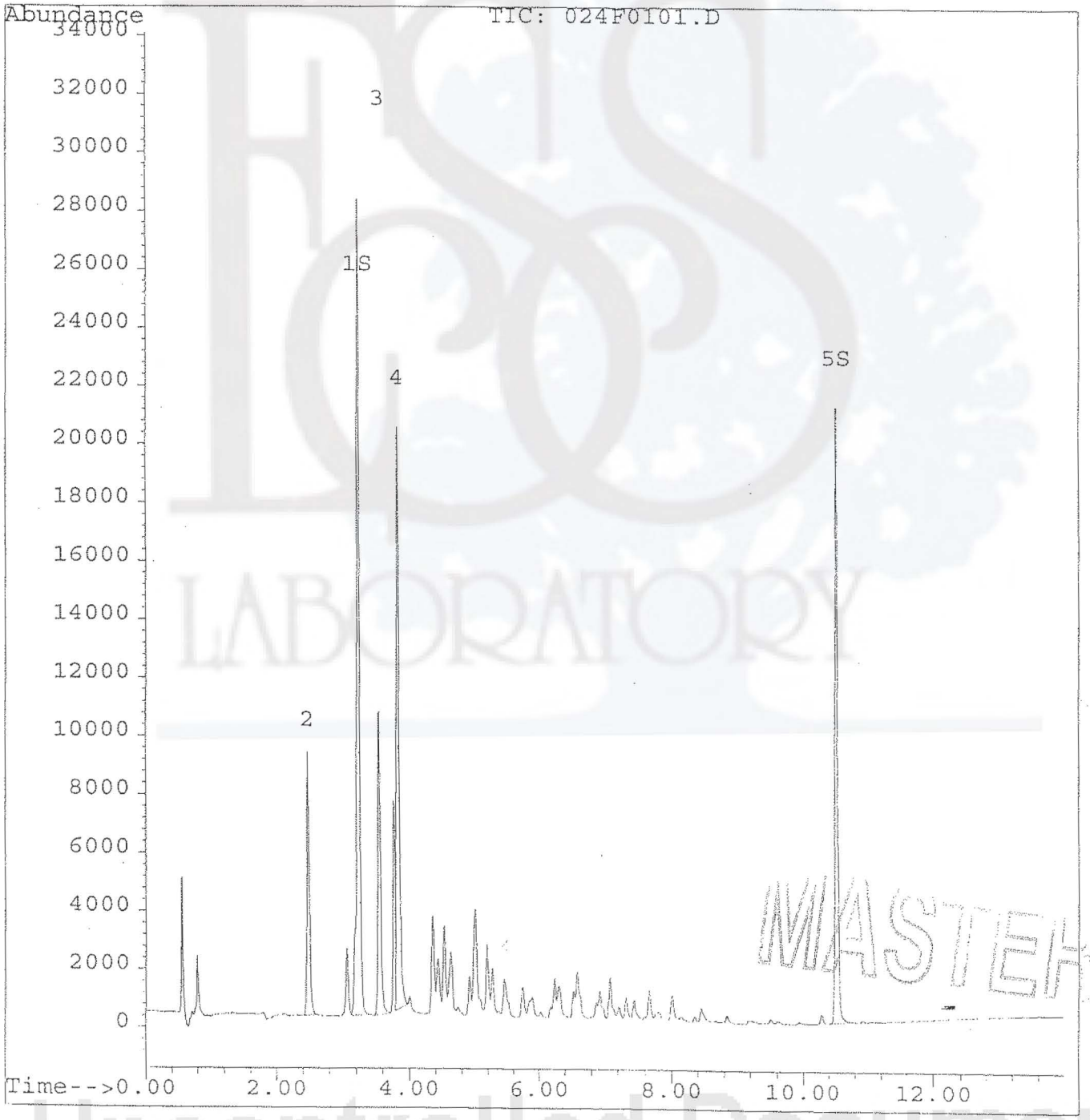
Quantitation Report

Data File : C:\HPCHEM\1\DATA\GE08209A\024F0101.D
Acq On : 20 Aug 98 09:50 PM
Sample : A21-3
Misc :
Quant Time: Aug 21 10:01 1998

Vial: 24
Operator: [GC]MS
Inst : GC 3
Multiplr: 1.00

Method : C:\HPCHEM\1\METHODS\A21F0820.M
Title :
Last Update : Fri Aug 21 10:04:00 1998
Response via : Multiple Level Calibration

Volume Inj. : 1 uL
Signal Phase : RTX-CLPESTICIDE
Signal Info : 0.53



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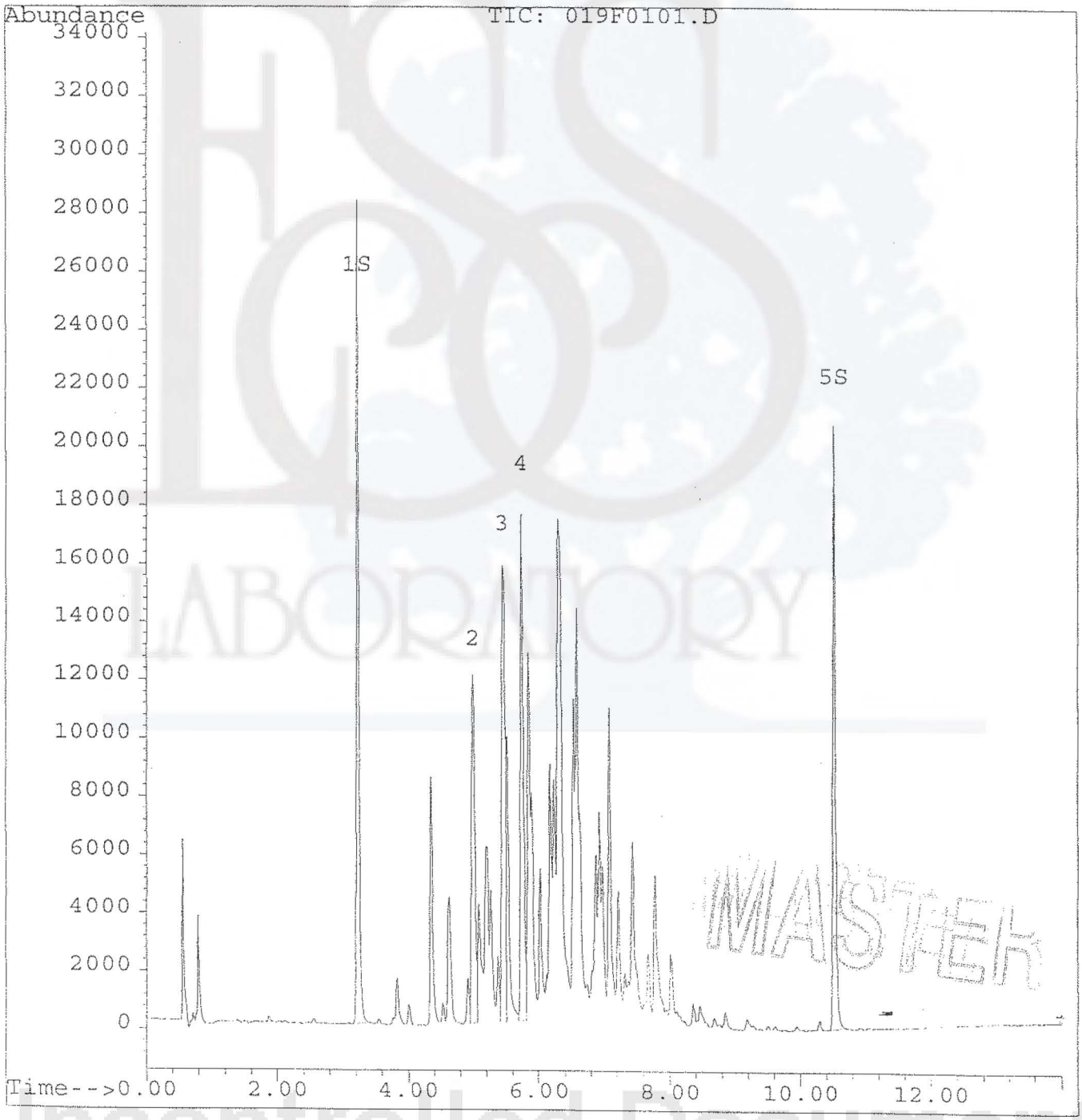
Quantitation Report

Data File : C:\HPCHEM\1\DATA\GE08209A\019F0101.D
Acq On : 20 Aug 98 08:27 PM
Sample : A48-3
Misc :
Quant Time: Aug 21 9:54 1998

Vial: 19
Operator: [GC]MS
Inst : GC 3
Multiplr: 1.00

Method : C:\HPCHEM\1\METHODS\A48F0820.M
Title :
Last Update : Fri Aug 21 09:56:52 1998
Response via : Multiple Level Calibration

Volume Inj. :
Signal Phase :
Signal Info :



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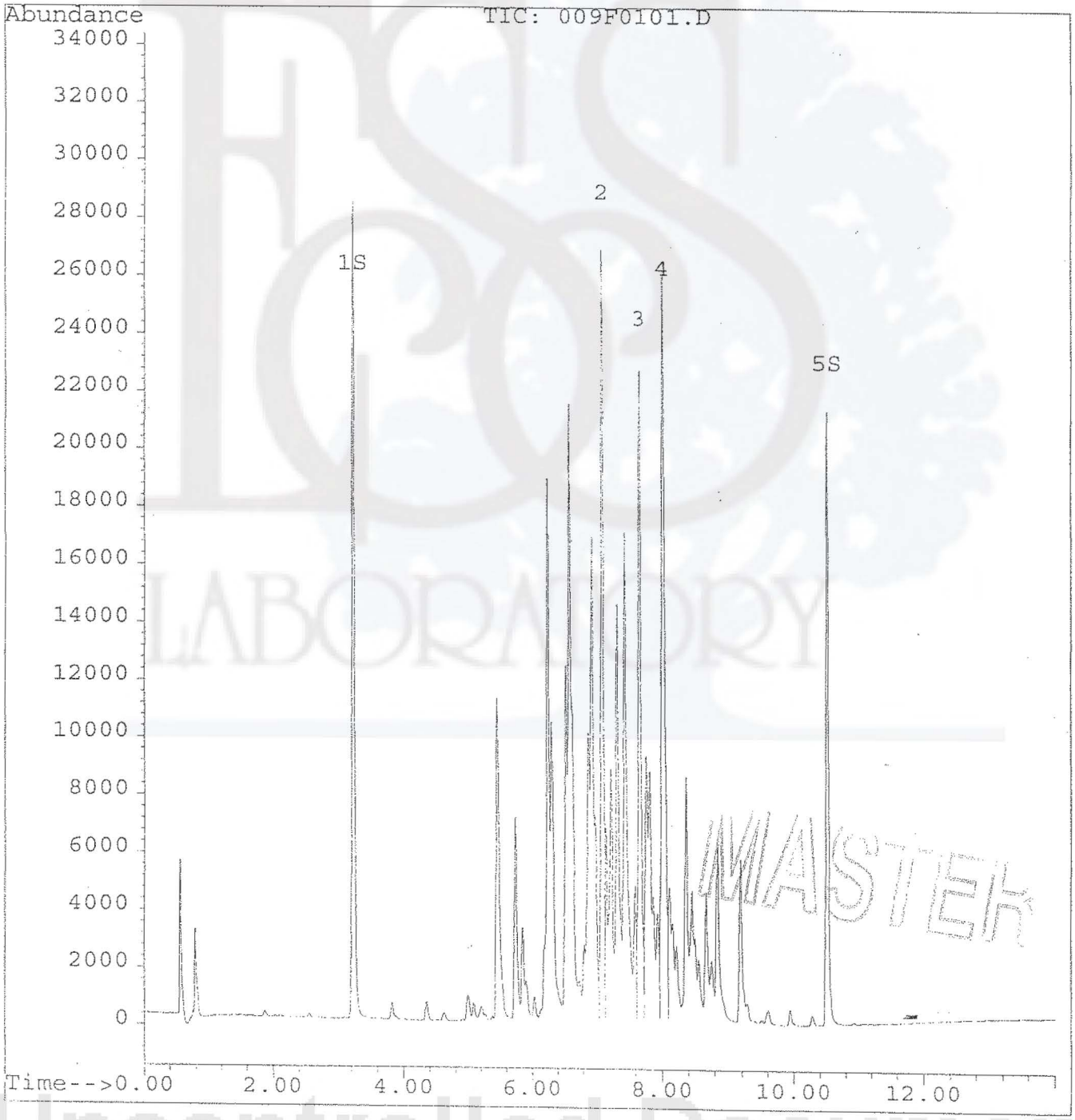
Quantitation Report

Data File : C:\HPCHEM\1\DATA\GE08209A\009F0101.D
Acq On : 20 Aug 98 05:41 PM
Sample : A54-3
Misc :
Quant Time: Aug 21 9:36 1998

Vial: 9
Operator: [GC]MS
Inst : GC 3
Multiplr: 1.00

Method : C:\HPCHEM\1\METHODS\A54F0813.M
Title :
Last Update : Fri Aug 21 09:37:37 1998
Response via : Multiple Level Calibration

Volume Inj. : 1 uL
Signal Phase : RTX-CLPESTICIDE
Signal Info : 0.53



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ATTACHMENT E

Summary of Method Quality Objectives for Method 8082 Polychlorinated Biphenyls

QC Element	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration (Non DOD work)	Instrument set up. Each time the ICV or CCV cannot meet criteria.	***Arochlor1016/1260*** • Minimum of 5 standards and contains all analytes • Low standard ≤ MRL • RSD≤20%, r≥0.995 (Do not force through zero). ***All other Arochlors*** • Single analysis at the reporting limit. • Evaluate quantitation at the low standard. Requanting the low standard should produce results within 70-130% of the true value.	• No allowance. Perform maintenance and recalibrate.
DOD REQUIREMENT Minimum five-point initial calibration (ICAL) for all analytes	ICAL prior to sample analysis	One of the options below Option 1: RSD for each analyte ≤ 20% Option 2: linear least squares regression: r ≥ 0.995 Option 3: non-linear regression: Coefficient of Determination (COD) $r^2 \geq 0.99$ (6 points shall be used for second order, 7 points shall ve used for third order).	• Problem must be corrected. No samples may be run until ICAL has passed. • Calibration may not be forced through the origin. • Quantitation for multicomponent analytes such as chlordane, toxaphene an Arochlors must be performed using a 5-point calibration. Results may not be quantitated using a single point
ICV – Second source verification standard	Immediately following initial calibration.	• %Rec = 80-120%. (DoD, MAMCP: 80-120%)	• If criteria are exceeded then remake and re-analyze ICV. If second consecutive ICV is within acceptable criteria then calibration is accepted, otherwise re-calibrate. (Option 2: re-analyze 2 consecutive CCV at 2 concentrations.)

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CCV	Prior to sample analysis, every 12 hours (10 field samples for DoD) and every 20 samples and at the end of each analytical sequence.	***Arochlor 1016/1260*** • Concentration level near midpoint of curve (DoD use 2 concentrations from Ical at or below midpoint, see 8.7.1) • Percent difference or percent drift must be 80-120%. (***DoD: all analytes within ±20% of the expected value from the ICAL***) ***All other Aroclors*** • Performed routinely for 1242/1254, all others must be analyzed within 12 hours of sample analysis.	• If criteria are exceeded then remake and re-analyze CCV. If second consecutive CCV is within criteria then calibration is verified, otherwise re-calibrate system and re-analyze any sample analyzed after the last valid CCV. Also, Option 2 from above is allowed. ***Exception: If CCV is exhibiting high bias (concentration is higher than upper limit) then any samples that are non-detect for that analyte may be reported.***
Method Blank	One per analytical batch of 20 or fewer samples.	• Matrix specific • Analytes < MRL (< ½ MRL for DoD)	• Report exceedance in the project narrative. • Any samples that are non-detect for that analyte may be reported. • Samples with concentrations that are 20x higher than the method blank may be reported. • Samples reported with a contaminated blank must be "B" flagged. • Re-extract if the above exceptions do not apply. If re-extract is within hold, report just the re-extracted data. If re-extract is outside hold then report both sets of data to client.
Blank spike/ Blank spike duplicate	One per analytical batch of 20 or fewer samples.	• Use standard source different than used for initial calibration • Concentration level should be between low and mid-level standard • Matrix specific • Percent recoveries between 40-140% and 30%RPD (see Table 1 for DoD). • Laboratories must develop in-house limits that are within above criteria.	• Report exceedance in the project narrative. • If the BS/BSD exhibits high bias and the samples are non-detect, then the samples may be reported with no corrective action needed. • Re-extract if the above exceptions do not apply. If re-extract is within hold, report just the re-extracted data. If re-extract is outside hold then report both sets of data to client.
Matrix Spike/ Matrix Spike duplicate	One per analytical batch of 20 or fewer samples	• Prepared using the same source as the blank spike • Concentration between low and mid-level standard • Matrix specific • Percent recoveries between 40-140. Laboratories should develop in-house limits.	• Check BS, if recoveries are acceptable then note exceedance in project narrative.

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		• RPD should be ≤ 50% • (MAMCP RPD <20 waters, <30 Soils) • See Table 1 for DoD/Navy/AFCEE limits	
Surrogates	Added to all samples and standards.	• Use a minimum of 2 surrogates. • Percent recovery of 30-150% *See Table 1 for DoD criteria.* • MCP requires that both columns and both surrogates be within criterion and reported.	• If the same surrogate is outside of limits on both columns, re-extract samples. If both surrogates are outside criteria on one column only, re-analyze extract • If a surrogate is diluted to a concentration below the lowest standard, then no corrective action is needed. • If surrogates are outside criteria for re-extract, report both sets of data. • If re-extract is within hold and within criteria, report just the re-extracted data. If re-extract is outside hold then report both sets of data to client. • Note exceedance in project narrative. If sample is not re-analyzed due to obvious interference (e.g., UCM), the chromatogram is to be included in the final report.
Identification and Quantitation		• Secondary column analysis: Laboratory must utilize a second dissimilar column to confirm positive pesticide results. • The lab must report the higher of the two results. • All QA/QC requirements must be met on secondary column as well. • Analytes must fall within retention time windows.	• Flag data that is >40% RPD between two columns. • IF high RPD can be attributed to interference on one of the two columns, the lab should report the lower value and provide a discussion in the case narrative that this approach was employed.

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Appendix F

Table V A-1: Specific QC Requirements and Performance Standards for PCBs (SW-846 8082A) Using WSC-CAM-V A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
Initial Demonstration of Proficiency	Laboratory Analytical Accuracy & Precision	(1) Must be performed prior to using method on samples. (2) Must be performed for each matrix. (3) Must contain Aroclors 1016/1260 for PCB Aroclor analysis and all target congeners for PCB congener analysis. (4) Must follow procedure in Section 8.4 of SW-846 8000B.	No	NA	Refer to Section 8.4 of SW-846 8000B and Section 1.1.2 of this protocol.	NA
Retention Time Windows	Laboratory Analytical Accuracy	(1) Prior to initial calibration and when a new GC column is installed. (2) Calculated according to the method (Section 7.6 of SW-846 8000B) (3) If acid cleanup is not performed, also analyze DDT/DDE/DDD standard.	No	NA	(1) For PCB Aroclor analysis, if interference is present for any of the Aroclor peaks used for quantitation with DDT, DDE, or DDD, either adjust GC conditions to obtain better resolution or choose another peak for the Aroclor of interest that does not coelute with DDT, DDE, or DDD. (2) For PCB congener analysis, if interference is present for any of the target congeners with DDT, DDE, or DDD, adjust GC conditions to obtain better resolution.	NA
Initial Calibration	Laboratory Analytical Accuracy	(1) Must be analyzed at least once prior to analyzing samples, when initial calibration verification or continuing calibration does not meet the performance standards, and when major instrument maintenance is performed. (2) Minimum of 5 standards (or 6 if non-linear regression used).	No	NA	(1) Recalibrate as required by method. (2) If recalculated concentrations from the lowest calibration standard are outside of 70-130% recovery range, either: * The RL limit must be	Sample analysis cannot proceed without a valid initial calibration. Report non-conforming compounds (%RSD >20, r <0.99, or r^2 <0.99) in laboratory narrative. If non-linear regression (e.g., quadratic equation) is used for calibration,

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Table V A-1: Specific QC Requirements and Performance Standards for PCBs (SW-846 8082A) Using WSC-CAM-V A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
		(3) *PCB Aroclors:* 5-point calibration with 1016/1260 required; 5-point calibration for other Aroclors may be warranted based on site-specific conditions (i.e., if nature of PCB contamination known). *Congeners:* 5-point calibration must include all target PCB congeners. (4) A minimum of 5 unique peaks must be evaluated for Aroclors 1016 and 1260. (5) Low standard must be ≤RL. (6) %RSD ≤20, r ≥0.99 (linear regression), or r^2 ≥0.99 (non-linear regression) for each PCB Aroclor or each PCB congener. (7) If %RSD >20, linear or non-linear regression must be used. (8) PCB Aroclors: For Aroclors which are not calibrated with 5-points, laboratory must perform single analysis of these Aroclors at the midpoint of the calibration curve. (9) Calibration must be performed under the same conditions as the samples. (10) If linear or non-linear regression used, verify the RL by recalculating concentrations in lowest calibration standard using the final calibration curve; recoveries must be 70-130%.			reported as an estimated value[2], or * The RL must be raised to the concentration of the next highest calibration standard that exhibits acceptable recoveries when recalculated using the final calibration curve.	this must be noted in the laboratory narrative along with the congeners or PCB Aroclors affected.
Initial Calibration Verification	Laboratory Analytical Accuracy	(1) Immediately after each initial calibration. (2) Concentration level near midpoint of curve. (3) Prepared using standard source different than used for initial	No	NA	Locate source of problem; recalibrate if either PCB Aroclor 1016/1260 or >10% of all PCB congeners are outside of criteria.	If recovery is outside of 80-120% for any PCB Aroclor or congener, report non-conformances in laboratory narrative.

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Table V A-1: Specific QC Requirements and Performance Standards for PCBs (SW-846 8082A) Using WSC-CAM-V A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
		calibration. (4) Must contain Aroclors 1016/1260 for PCB Aroclor analysis and all target congeners for PCB congener analysis. (5) Percent recoveries must be between 80-120% for each PCB Aroclor or congener.				
Continuing Calibration	Laboratory Analytical Accuracy	(1) Prior to samples, every 12 hours or every 20 samples, whichever is more frequent, and at the end of the analytical sequence. (NOTE: if internal standard calibration used, the continuing calibration at the end of the analytical sequence is not required). (2) Concentration level near midpoint of curve. (3) *PCB Aroclors:* Must contain Aroclors 1016/1260. Aroclors other than 1016/1260 must be verified with a one-point standard within 12 hours of being detected in a sample. *Congeners:* Must include all target PCB congeners. (4) %D must be ≤20 for each PCB Aroclor or PCB congener. (5) Verify that all analytes fall within retention time windows. (6) Area count of internal standard in continuing calibration must be within ±50% of the average area count in the associated initial calibration.	No	NA	(1) Perform instrument maintenance, reanalyze continuing calibration and/or recalibrate as required by method. (2) Renalyze "associated samples" if beginning or ending continuing calibration exhibited low response. (3) Reanalyze "associated samples" if beginning or ending continuing calibration exhibited high response and associated PCB Aroclors or congeners were detected in the "associated samples." NOTE: "Associated samples" refers to all samples analyzed since the last acceptable continuing calibration.	Report non-conforming compounds (%D >20) and associated samples in laboratory narrative.
Method Blank	Laboratory Method Sensitivity (contamination evaluation)	(1) Extracted with every batch or every 20 samples, whichever is more frequent. (2) Matrix-specific (e.g., water, soil).	Yes	NA	(1) If concentration of contaminant in sample is <10x concentration in blank, locate source of	(1) If sample re-extraction is not possible, report non-conformance in

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Table V A-1: Specific QC Requirements and Performance Standards for PCBs (SW-846 8082A) Using WSC-CAM-V A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
		(3) Target analytes must be <RL.			contamination; correct problem; re-extract and re-analyze method blank and associated samples. (2) No corrective action required if concentration of contaminant in sample is >10x concentration in blank or if contaminant not detected in sample.	laboratory narrative. (2) If contamination of method blanks is suspected or present, the laboratory, using a "B" or some other convention, should qualify the sample results. Blank contamination should also be documented in the laboratory narrative. (3) If re-extraction is performed within holding time and yields acceptable method blank results, the laboratory may report results of the re-extraction only. (4) If re-extraction is performed outside of holding time, the laboratory must report results of both the initial extraction and re-extraction.

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Table V A-1: Specific QC Requirements and Performance Standards for PCBs (SW-846 8082A) Using WSC-CAM-V A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
Laboratory Control Sample (LCS)	Laboratory Analytical Accuracy	(1) Extracted with every batch or every 20 samples, whichever is more frequent. (2) Concentration level near midpoint of curve. (3) *PCB Aroclors:* 1016/1260 required. Optionally, LCSs may be spiked with other Aroclors which have been fully calibrated, based on site-specific conditions (i.e., if specific Aroclors are known to be present or expected in samples). *Congeners:* Must include all target PCB congeners. (4) Matrix-specific (e.g., soil, water). (5) Percent recoveries must be between 40-140% for target analytes. (6) Must be prepared in a water-miscible solvent (e.g., acetone, methanol).	Yes	Recovery <10%; affects nondetect results for affected PCB Aroclor or PCB congener in all samples extracted with this LCS.	(1) Locate source of problem; re-extract and re-analyze LCS and associated samples if either Aroclor 1016/1260 or >10% of all PCB congeners are outside of criteria. (2) If ≤10% of PCB congeners are outside of the acceptance criteria, re-extraction is not required as long as recoveries are >10%. (3) If >10% of PCB congeners or either Aroclor 1016/1260 are above the acceptance criteria (>140), reextraction is not required if the affected congeners or all PCB Aroclors were not detected in associated samples.	(1) If sample re-extraction is not possible, report nonconformance in laboratory narrative. (2) If recovery is outside of 40-140% for any PCB Aroclor or congener, report non-conforming compounds in laboratory narrative. (3) If re-extraction is performed within holding time and yields acceptable LCS results, the laboratory may report results of the re-extraction only. (4) If re-extraction is performed outside of holding time, the laboratory must report results of both the initial extraction and re-extraction.
LCS Duplicate	Laboratory Analytical Accuracy & Precision	(1) Extracted with every batch or every 20 samples, whichever is more frequent. (2) Concentration level near midpoint of curve. (3) *PCB Aroclors:* 1016/1260 required. Optionally, LCS Duplicates may be spiked with other Aroclors which have been fully calibrated, based on site-specific conditions (i.e., if specific Aroclors are known to be present or expected in samples).	Yes	Recovery <10%; affects nondetect results for affected PCB Aroclor or PCB congener in all samples extracted with this LCS.	(1) Locate source of problem; re-extract and re-analyze LCS and associated samples if either Aroclor 1016/1260 or >10% of all PCB congeners are outside of recovery acceptance criteria. (2) If ≤10% of PCB congeners are outside of the recovery acceptance	(1) If sample re-extraction is not possible, report nonconformance in laboratory narrative. (2) If recovery is outside of 40-140% for any PCB Aroclor or congener or if RPD is outside of criteria, report non-conforming compounds in laboratory

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Table V A-1: Specific QC Requirements and Performance Standards for PCBs (SW-846 8082A) Using WSC-CAM-V A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
		Congeners: Must include all target PCB congeners. (4) Matrix-specific (e.g., soil, water). (5) Percent recoveries must be between 40-140% for target analytes. (6) RPDs must be ≤20 for waters and ≤30 for solids. (7) Must be prepared in a water-miscible solvent (e.g., acetone, methanol).			criteria, re-extraction is not required as long as recoveries are >10%. (3) If >10% of PCB congeners or either Aroclor 1016/1260 are above the recovery acceptance criteria (>140%), reextraction is not required if the affected congeners or all PCB Aroclors were not detected in associated samples.	narrative. (3) If re-extraction is performed within holding time and yields acceptable LCS results, the laboratory may report results of the re-extraction only. (4) If re-extraction is performed outside of holding time, the laboratory must report results of both the initial extraction and re-extraction.
MS/MSD	Method Accuracy & Precision in Sample Matrix	(1) Every 20 samples (at discretion of laboratory or at request of data user). (2) Matrix-specific. (3) Concentration level near midpoint of curve. (4) *PCB Aroclors:* 1016/1260 required. Optionally, MS/MSDs may be spiked with other PCB Aroclors which have been fully calibrated, based on site-specific conditions (i.e., if specific Aroclors known to be present or expected in samples). *Congeners:* Must include all target PCB congeners. (5) Percent recoveries between 40 – 140%. (6) RPDs ≤20 for waters and ≤30 for solids. (7) Must be prepared in a water-miscible solvent (e.g., acetone, methanol).	Yes ONLY when requested by the data user	Recovery <10%; affects nondetect result for affected PCB Aroclor or PCB congener in unspiked sample only.	Check LCS; if recoveries are acceptable in LCS, narrate non-conformance.	Note exceedances in laboratory narrative.

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Table V A-1: Specific QC Requirements and Performance Standards for PCBs (SW-846 8082A) Using WSC-CAM-V A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
Surrogates	Method Accuracy in Sample Matrix	(1) Minimum of 2 surrogates, one that elutes at beginning of GC run and one that elutes at end of GC run. Recommended surrogates: *PCB Aroclor analysis:* TCMX and DCB *PCB Congener analysis:* TCMX or DBOFB and BZ198 (2) Percent recoveries must be between 30-150% for both surrogates on both columns.	Yes (report surrogate recoveries from both columns)	Recovery <10%; affects all nondetect results in affected sample.	If the same surrogate is outside limits on both columns: (1) Re-extract the sample if surrogate recoveries are low and there is no chromatographic interference. (2) Re-extract the sample if surrogate recoveries are high and PCB Aroclors or PCB congeners were detected in the sample. NOTES: (a) If surrogate recoveries are high and target analytes are not detected in sample, re-extraction is not required. (b) If chromatographic interference is present and surrogate recovery would cause rejection of data (i.e., <10%), reanalyze sample on dilution. (c) If a surrogate is diluted to a concentration below that of the lowest calibration standard, reextraction and/or reanalysis is not required.	(1) Report recoveries outside of 30-150% in laboratory narrative. (2) If re-extraction yields similar surrogate non-conformances, the laboratory must report results of both the initial extraction and re-extraction. (3) If re-extraction is performed within holding time and yields acceptable surrogate recoveries, the laboratory may report results of the re-extraction only. (4) If re-extraction is performed outside of the holding time and yields acceptable surrogate recoveries, the laboratory must report results of both the initial extraction and re-extraction. (5) If sample is not re-extracted due to chromatographic interference, the laboratory must provide the chromatogram in the data report.
Internal Standards (Congeners only)	Laboratory Analytical Accuracy and Method Accuracy in	(1) Minimum of 1. Recommended internal standard: DCB (2) Area counts in samples must be between 50 – 200% of the area counts	No	Recovery <20%; affects all nondetect results quantitated using	If internal standard is outside of limits, reanalyze sample unless chromatographic	(1) Report non-conformances in laboratory narrative. Include actual recovery

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Table V A-1: Specific QC Requirements and Performance Standards for PCBs (SW-846 8082A) Using WSC-CAM-V A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
	Sample Matrix	in the associated continuing calibration standard. (3) Retention times of internal standards must be within ±30 seconds of retention times in associated continuing calibration standard.		affected internal standard in associated sample.	interference present. NOTE: If chromatographic interference is present and internal standard area would cause rejection of data (i.e., <20%), reanalyze sample on dilution.	of internal standard and provide summary of analytes quantitated using the internal standard. (2) If reanalysis yields similar internal standard non-conformances, the laboratory must report results of both analyses. (3) If reanalysis is performed within holding time and yields acceptable internal standard recoveries, the laboratory may report results of the reanalysis only. (4) If reanalysis is performed outside of the holding time and yields acceptable internal standard recoveries, the laboratory must report results of both analyses. (5) If sample is not reanalyzed due to chromatographic interference, the laboratory must provide the chromatogram in the data report.
Identification and Quantitation	NA	(1) Peak area is the expected default to be used for quantitation of PCB Aroclors and congeners under most circumstances. Regardless if peak area or peak height is used, the same method used for quantitation of	NA	If RPD >100 for PCB congener, reject positive result for affected PCB congener. If RPD >500 for	If the RPD between the dual column results is >100 for PCB congeners or >500 for PCB Aroclors, reanalyze the sample on dilution. Both analyses	If the RPD between the dual column results exceeds 40, the laboratory must qualify the sample results and/or note the

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Table V A-1: Specific QC Requirements and Performance Standards for PCBs (SW-846 8082A) Using WSC-CAM-V A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
		samples must also be used for calibration standards. (2) *PCB Aroclors:* The laboratory must quantitate all Aroclors with the same five peaks used for calibration. If interference exists with select peaks, these peaks do not have to be included in the quantitation of the Aroclor; however, a minimum of three peaks is required. All peaks must be ≥25% of the height of the largest PCB Aroclor peak. At least one peak must be unique to the PCB Aroclor. (3) *PCB Congeners:* The laboratory must use the average calibration factor, response factor, linear or non-linear regression curve generated from the associated initial calibration for quantitation of each PCB congener. *PCB Aroclors:* Laboratory should use the average calibration factor, linear or non-linear regression curve for each of three to five peaks from each concentration level to quantitate Aroclors 1016 and 1260. Laboratory should use the average calibration factor for each of three to five peaks from single point standard to quantitate remaining Aroclors (when only single-point standard analyzed). If 5-point calibration is performed for other Aroclors, follow procedure for 1016 and 1260. Calculate concentration of Aroclor using each individual peak and calculate the average concentration of the three to five results to obtain the final Aroclor concentration.		PCB Aroclor, reject positive result for affected PCB Aroclor.	must be reported. Alternatively, additional sample cleanup techniques may be warranted.	exceedance in the laboratory narrative. If the RPD exceedance is due to interference, the lower of the dual column values can be reported; this must be noted in the laboratory narrative.

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Table V A-1: Specific QC Requirements and Performance Standards for PCBs (SW-846 8082A) Using WSC-CAM-V A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
		(4) Secondary column analysis: Laboratory must utilize a second dissimilar column to confirm positive results. The laboratory must report the higher of the two results. All required QC parameters (e.g., calibrations, LCSs, etc.) must be met on the secondary column as well. (5) Results must be reported with 2 or more "significant figures" if ≥ RL. If reporting values below the RL, report with 1 or more "significant figures".[3]				
General Reporting Issues	NA	(1) The laboratory must only report values ≥ the sample-specific reporting limit. (2) Dilutions: If diluted and undiluted analyses are performed, the laboratory should report results for the lowest dilution within the valid calibration range for each analyte. The associated QC (e.g., method blanks, surrogates, etc.) for each analysis must be reported. **NOTE: Laboratories shall not perform dilutions on samples due to sulfur interference. Laboratories must employ a cleanup technique to reduce the presence of sulfur interference.** **It is highly recommended that acid cleanup be performed on all sample extracts prior to analysis.** (3) Results for soils/sediments must be reported on a dry-weight basis for comparison to MCP regulatory standards. (4) Refer to Appendix V A-1 for chain-of-custody requirements regarding preservation, cooler temperature, and	NA	NA	NA	(1) Complete analytical documentation for diluted and undiluted analyses must be made available for review during an audit. (2) The performance of dilutions must be documented in the laboratory narrative or on the report form. Unless due to elevated concentrations of target compounds, reasons for dilutions must be explained in the laboratory narrative. (3) If PCB Aroclors are not detected but chromatogram shows evidence of weathered Aroclors or potential presence of PCBs, this must be noted in the laboratory narrative and a copy of the

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Table 2

Summary of Method Quality Objectives for Method 8082
Polychlorinated Biphenyls

QC Element	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration (Non DOD work)	Instrument set up. Each time the ICV or CCV cannot meet criteria.	***Arochlor1016/1260*** • Minimum of 5 standards and contains all analytes • Low standard ≤ MRL • RSD≤20%, r≥0.995 (Do not force through zero). ***All other Arochlors*** • Single analysis at the reporting limit.	• No allowance. Perform maintenance and recalibrate.
DOD REQUIREMENT Minimum five-point initial calibration (ICAL) for all analytes	ICAL prior to sample analysis	One of the options below Option 1: RSD for each analyte ≤ 20% Option 2: linear least squares regression: r ≥ 0.995 Option 3: non-linear regression: Coefficient of Determination (COD) $r^2 \geq 0.99$ (6 points shall be used for second order, 7 points shall ve used for third order).	• Problem must be corrected. No samples may be run until ICAL has passed. • Calibration may not be forced through the origin. • Quantitation for multicomponent analytes such as chlordane, toxaphene an Arochlors must be performed using a 5-point calibration. Results may not be quantitated using a single point
ICV – Second source verification standard	Immediately following initial calibration.	• %Rec = 85-115%. (DoD/Navy/AFCEE 80-120%)	• If criteria are exceeded then remake and re-analyze ICV. If second consecutive ICV is within acceptable criteria then calibration is accepted, otherwise re-calibrate. (Option 2: re-analyze 2 consecutive CCV at 2 concentrations.)

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CCV	Prior to sample analysis, every 12 hours (10 field samples for DoD/ Navy/ AFCEE) and every 20 samples and at the end of each analytical sequence.	***Arochlor 1016/1260*** • Concentration level near midpoint of curve (Navy/DoD use 2 concentrations from Ical at or below midpoint, see 8.7.1) • Percent difference or percent drift must be 85-115%. (***USACE: grand mean <15% with no individual analyte>30%; DoD/Navy:all analytes within ±20% of the expected value from the ICAL; AFCEE: 20%D***) ***All other Aroclors*** • Performed routinely for 1242/1254, all others must be analyzed within 12 hours of sample analysis.	• If criteria are exceeded then remake and re-analyze CCV. If second consecutive CCV is within criteria then calibration is verified, otherwise re-calibrate system and re-analyze any sample analyzed after the last valid CCV. Also, Option 2 from above is allowed. ***Exception: If CCV is exhibiting high bias (concentration is higher than upper limit) then any samples that are non-detect for that analyte may be reported.***
Method Blank	One per analytical batch of 20 or fewer samples.	• Matrix specific • Analytes < MRL (< ½ MRL for USACE/Navy/ DoD/AFCEE)	• Report exceedance in the project narrative. • Any samples that are non-detect for that analyte may be reported. • Samples with concentrations that are 20x higher than the method blank may be reported. • Samples reported with a contaminated blank must be "B" flagged. • Re-extract if the above exceptions do not apply. If re-extract is within hold, report just the re-extracted data. If re-extract is outside hold then report both sets of data to client.
Blank spike/ Blank spike duplicate	One per analytical batch of 20 or fewer samples.	• Use standard source different than used for initial calibration • Concentration level should be between low and mid-level standard • Matrix specific • Percent recoveries between 40-140% and 30%RPD (USACE % Recovery 50-130, see Table 1 for Navy/DoD/AFCEE). • Laboratories must develop in-house limits that are within above criteria.	• Report exceedance in the project narrative. • If the BS/BSD exhibits high bias and the samples are non-detect, then the samples may be reported with no corrective action needed. • Re-extract if the above exceptions do not apply. If re-extract is within hold, report just the re-extracted data. If re-extract is outside hold then report both sets of data to client.
Matrix Spike/ Matrix Spike	One per analytical batch of 20 or fewer samples	• Prepared using the same source as the blank spike • Concentration between low and mid-level standard	• Check BS, if recoveries are acceptable then note exceedance in project narrative.

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ESS Laboratory
Division of Thielsch Engineering
Cranston, RI

SOP 60_8100-mod
Total Petroleum Hydrocarbons
(SW846 Method 8100 modified/Conn ETPH)
BY GC/FID

REVIEWED BY:

Operations Manager	Date 6/27/08
QA Manager	Date 6/23/08
Laboratory Director	Date 6/28/08

MASTER

Master marked in red. Controlled copies marked in green.

Total Petroleum Hydrocarbons (SW846 Method 8100 modified/Conn ETPH)

1.0 SCOPE AND/OR APPLICATION

1.1 This method has been modified to determine the total petroleum hydrocarbon (TPH) content of samples from C_9-C_{36}. This range represents the major components of a number of petroleum products such as kerosene, jet and diesel fuel, No. 2 to 6 fuel oils and motor oils. Gasoline range organics are not quantitated by this method.

1.2 Compounds less than C_{36} present in products such as motor oils or lubricating oils can be quantitated using the calibration curve generated in this method. However, compounds greater than C_{36} must be reported separately from TPH and must be qualified as estimated. Components lighter than C_9 in products such as gasoline are also detectable but may be completely or partially lost during sample preparation.

1.3 The Reporting limits commonly used are as follows for the applicable matrices (preparative methods are listed in section 2.0):

Matrix	Weight Vol	Final Extract Vol	MRL
Water	1.0 liter	1.0 ml	200 ppb
Soil	20-30 g	1.0 ml	37.5-25 ppm
Waste Oil	1.0 g	10 ml	7500 ppm

2.0 Method Summary

2.1 This TPH method provides gas chromatographic conditions for the detection of total petroleum hydrocarbons in the C_9-C_{36} range. This procedure is based on a solvent extraction followed by analysis on a high-resolution capillary column gas chromatograph with duel flame ionization detectors. Sample preparation is discussed separately in the respective preparation SOP.

2.2 A capillary column and temperature program is used in the gas chromatograph to separate the organic compounds and a Flame Ionization Detector (FID) achieves detection.

2.3 Samples can be prepared by one of the following extraction methods, refer to the appropriate SOP:

Matrix	Method #	Method Summary
Water	3510	Separatory Funnel Liquid-Liquid Extraction
Soil	3541	Automated Soxhlet Extraction
Soil	3546	Microwave Extraction
Oil	3580	Waste Dilution

UNCONTROLLED

3.0 HEALTH AND SAFETY

3.1 Each employee has been trained and has acknowledged being trained in the safe use and handling of chemicals being used in the laboratory. This training has been performed according to the ESS Training SOP 80_0016 and by the Chemical Hygiene Plan SOP 90_0001 in conjunction with the Safety orientation

3.2 To minimize exposure, process samples in an exhaust hood or well-ventilated workspace. When working with samples and chemicals, wear gloves to minimize contact and possible absorption. Always wear appropriate eye protection.

3.3 Proper emergency response to spills or injury should be reviewed by the laboratory employee prior to attempting this procedure. This includes location of spill kits, emergency eyewash and showers, fire fighting equipment, as well as evacuation routes.

3.4 Material Safety Data Sheets are available for all chemicals used in this procedure. All laboratory employees are required to read these before handling these chemicals.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

4.1 Aqueous samples are collected in 1 liter amber glass bottles with Teflon-lined screw caps. Aqueous samples are preserved to a pH of <2 with 1:1 HCl and cooled to 4°C immediately after collection.

4.2 Soil and sediment samples are collected in 4 oz (120 ml) wide-mouth glass jars with Teflon-lined screw caps and cooled to 4°C immediately after collection.

4.3 Aqueous samples must be extracted within 7 days of collection, and analyzed within 40 days of extraction.

4.4 Soil and sediment samples must be extracted within 14 days of collection, and analyzed within 40 days of extraction.

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

5.1 Method interferences are reduced by washing all glassware with hot soapy water and then rinsing with warm tap water, acetone or methanol, and methylene chloride.

5.2 High purity reagents must be used to minimize interference problems.

5.3 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. Whenever an unusually concentrated sample is analyzed, it must be followed by the analysis of a system solvent blank to check for cross-contamination.

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5.4 To minimize mass discrimination, the injection system must be cleaned on a regular basis. Loss of hydrocarbon C_{28} – C_{36} response can occur after running highly contaminated samples.

5.5 C36 response is greatly affected by the length of column inserted into the injection port. To meet Connecticut criteria insert a column length of 4-5 mm.

6.0 APPARATUS AND MATERIALS

6.1 Gas Chromatograph HP 5890 Series II.

6.2 HP7673 Autosamplers.

6.3 Dual FID (Flame Ionization Detector).

6.4 Column DB-5 (J&W) 30m * 0.25 mm ID * 0.5 um film thickness or equivalent

6.5 Vials.

6.5.1 Sample vials with Teflon lined crimp tops.

6.5.2 14 ml glass vials with Teflon lined caps.

6.6 Volumetric flasks 10 ml-100 ml.

6.7 Microsyringes 10 µl, 100 µl , 250 µl, 500 µl, 1000 µl.

6.8 **Data system:**

6.8.1 **Computer:** The Semi-Volatiles laboratory has one GC/FID system analyzing method 8100-mod. SVOA GC2 has an AST computer with a Windows 95 operating system. All computer systems are networked to a Windows 2003 server, which is the destination of all files. A differential back-up is performed nightly and a full back is performed each weekend using Symantec Backup Exec to tapes. As the systems acquires and stores data onto the server, the server becomes full. The data is downloaded and archived onto an external hard drive.

6.8.2 **Software:** HP/Agilent Environmental Chemstation - The software is interfaced to the flame ionization detectors and allows the continuous acquisition and storage on machine-readable media of all chromatograms obtained throughout the duration of the instrument program. The software is capable of integrating the abundance in any EICP between specified times. Current versions G1045A version 01.00.

7.0 REAGENTS AND STANDARDS

7.1 **Reagents**

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7.1.1 Reagent Water: organic free water (ASTM Type I reagent grade water).

7.1.2 Solvents: methylene chloride and acetone methanol; pesticide grade or better. Store away from other solvents.

7.1.3 Sodium sulfate: (ACS) granular, anhydrous.

7.1.4 Ottawa and/or masonry sand: free of extractable petroleum hydrocarbons.

7.2 **Standards**: Store *all* standard solutions (primary, stock, working, and surrogate) at room temperature in the dark.

7.2.1 **Primary Standards**: Unopened primary standards are not kept beyond the manufacturer's expiration date and, once opened, primary standards are not kept beyond six months, or the manufacturer's expiration date (whichever comes first).

Solution name	Concentration	Vendor	Cat #
Aliphatic Hydrocarbon Standard	1000 µg/ml	Ultra	SMA-310
Aliphatic Hydrocarbon Standard (Second source)	1000 µg/ml	Accustandard	*
o-Terphenyl	2000 µg/ml	Ultra	IST-480

* Proposed Mass DEP – Aliphatic Hydrocarbons DRH-007S

All solutions are stored at room temperature.

7.2.2 **Calibration Stock Standard:** This calibration standard is prepared by making a 1:2 dilution of the primary standard. Five ml of the primary aliphatic hydrocarbon standard and 2.5 ml of the o-Terphenyl primary standard is diluted to 10 ml in a 10 ml class A volumetric with methylene chloride. The final concentration for these standards will be 500 mg/L. The following analytes are in this mix:

Carbon #		Carbon #		Carbon #	
9	n-Nonane	**18**	n- Octodecane	**26**	n- Hexacosane
10	n-Decane	**19**	n-Nonadecane	**28**	n-Octacosane
12	n- Dodecane	**20**	n-Eicosane	**30**	n- Triacontane
14	n-Tetradecane	**22**	n- Docosane	**36**	n-Hexatriacontane
16	n- Hexadecane	**24**	n- Tetracosane		

o-Terphenyl

WASTED

7.2.3 **Working Calibration Standards**

7.2.3.1 The lower linear range of the system is defined as the collective response of the 14 aliphatic compounds (70-ppm). The upper linear range for the collective C_9-C_{36} aliphatics is defined by peak height measurement based upon the maximum peak height documented for

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the 500-ppm aliphatic working standard. If the 500 µg/ml standard is not used for calibration then the 250 µg/ml standard will define the linear dynamic range

7.2.3.2 Prepare working solutions for initial and daily calibrations by transferring the following volumes of stock solution using Methylene Chloride as a solvent.

Cal Stock Conc.	Vol. added	Final Vol.	Final Aliph.Conc.	C_9-C_{36} Conc.
500 ug/ml	1000 ul	1.0 ml	500 ug/ml	7000
500 ug/ml	500 ul	1.0 ml	250 ug/ml	3500
500 ug/ml	200 ul	1.0 ml	100 ug/ml (CCV)	1400
500 ug/ml	100 ul	1.0 ml	50 ug/ml	700
500 ug/ml	20 ul	1.0 ml	10 ug/ml	140
500 ug/ml	10 ul	1.0 ml	5 ug/ml	70
10 ug/ml	100 ul	1.0 ml	1 ug/ml	14

7.2.4 **Second Source calibration standards**- A calibration stock standard (500 ug/ml) and working initial calibration verification standard (100 ug/ml) are prepared in the same manner as the calibration standards.

8.0 PROCEDURE

8.1 Instrument Set-Up

8.1.1 All daily maintenance, as recommended by the instrument manufacturer, must be performed on the GC before analysis can be run. This must be recorded in the maintenance Logbook. See section 18.0 for maintenance and troubleshooting.

8.1.2 The chromatographic conditions have been optimized using a high resolution capillary column with a slow ramp rate from 50°C to 310°C. The total run time is over 25 minutes. The resulting chromatogram provides extensive chromatographic information covering a wide range of materials including, but not limited to, light and medium fuel oils, heavy oils (such as crude oils), coal tar, and lubricating oils.

8.1.3 The column must resolve diesel components and the solvent front from C_9.

8.1.4 Settings

	Injectors A and B
Injection port temperature	290°C
Detector temperature	300°C
Injection volume	1-2 ul
Recommended carrier gas H2 :	0.5 to 1.5 ml/min
Air:	400 ml/min

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Hydrogen:	35 ml/min

8.2 Instrument Calibration

8.2.1 ESS Laboratory's policy is that the audit trail on the Chemstation/Enviroquant software is always on. This ensures that any changes made to the instrument operating method be documented through the audit trail.

8.2.2 Calibrate the GC with five to seven calibration working standards. The GC System is calibrated using the external standard method.

8.2.3 Prepare Aliphatic Hydrocarbon calibration standards at a minimum of five concentration levels, see 7.2.3.2.

8.2.4 Inject each calibration standard using the technique that will be used to introduce the actual samples into the gas chromatograph (e.g., 2 µl injections).

8.2.5 Tabulate the area responses against the mass of TPH injected. This applies to the individual aliphatic compounds as well as the C_9-C_{36} range. The C_9-C_{36} retention time (Rt) window is defined as the Rt of n-Nonane (C_9) to the Rt of n-Hexatriacontane (C_{36}). The results are used to prepare a calibration curve for the individual aliphatic compounds, as well as the TPH range. The ratio of the response to the amount injected, defined as the calibration factor (CF), is calculated at each standard concentration using Equation 1. This is accomplished through the EnviroQuant software. ***After generating the initial calibration curve in Enviroquant, the analyst must visually check that each calibration standard was entered into the new calibration method. This is accomplished by checking that the area response for one compound or range from each calibration standard's printout corresponds to the area count listed in the calibration method in Enviroquant.***

Equation 1

$$\textbf{Calibration Factor (CF)} = \frac{(A_s)}{(C_s)}$$

Where:

A_s = Area for TPH in the standard (C_9-C_{36} range or individual aliphatic compounds).

C_s = Mass of TPH injected, ng.

8.2.5.1 If the percent relative standard deviation (%RSD) of the calibration factors is equal to or less than 20% for the calibration standards, as determined using Equation 2, linearity through the origin can be assumed. Refer to Tables 1 & 2 in Section 10.4.

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8.2.5.2 If the system does not meet this standard, then a linear regression calibration curve may be used. If linear regression is used, then at a minimum, the correlation coefficient, r, must be $\geq$ 0.99.

8.2.5.3 Any individual standard can be omitted. A minimum of five points are needed to satisfy method requirements. Omission of a point will redefine the calibration range. Dilutions will be made accordingly.

8.2.5.4 If the system does not meet these standards then the calibration data is not acceptable. If the calibration does not pass the system may need to be maintained and a new calibration performed.

Equation 2: **Percent Relative Standard Deviation**

$$\% \text{ RSD} = \frac{\text{Stand Dev of 5 CFs} \times 100\%}{\text{Mean of 5 CFs}}$$

8.2.6 **Initial Calibration verification standard (ICV)** – Immediately following generation of an initial calibration a second source standard mid level calibration is run after each initial calibration. The concentration of the individual aliphatic compounds as well as the TPH C_9-C_{36} range in the second source must be within 20 %Difference/Drift of the expected value. Percent Drift is used when linear regression is used for the initial calibration. %Drift is calculated as %recovery of ICV value versus actual concentration. See Section 10 for method-specific exceptions. Percent Difference is calculated as follows:

Equation 3:

$$\textbf{Percent Difference } (\%D) = \frac{R_2 - R_1}{R_1} \times 100\%$$

Where:

$R_1 = \overline{CF}$ from the calibration (average calibration factor)
R_2 = CF from the check standard

8.2.7 **Continuing Calibration Verification:** At a minimum, the CF of the $C_9 - C_{36}$ TPH range and individual aliphatic compounds must be verified on each working day prior to sample analysis. It must also be verified after every 20 samples (10 field samples for DoD/Navy/AFCEE) or 12 hours. This is performed by the injection of a mid-level calibration standard to verify instrument performance and linearity. If the percent difference/drift (%D) for the TPH range and individual aliphatic compounds varies from the predicted response by more than ±20%, as calculated using Equation 3. See corrective action in Section 10.0 for CCVs that are outside criterion. **(The CCV concentration, when performing DoD/Navy work must be at a minimum two different concentrations throughout the analytical run. One standard must be at or below the mid-range standard.)** ESS Lab

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alternates between the 50 ppm and 100 ppm standards. Refer to Tables 1 & 2 in Section 10.4.

8.2.8 Record continuing calibration acceptability, if applicable, on attachment A.

8.3 SAMPLE ANALYSIS

8.3.1 Prior to running samples, a midlevel calibration standard must be analyzed to verify the curve (8.2.6). This calibration standard is analyzed under the same conditions as the calibration curve. Once the instrument has a valid continuing calibration, sample analysis may begin.

8.3.2 State of Connecticut ETPH Mass Discrimination protocol requires that the first CCAL standard in a sequence be evaluated for mass discrimination. The response factor from each alkane (C_9-C_{36}) is compared to the average response factor of all the alkanes in the initial calibration. An individual alkane response factor cannot drift more than 20% from the average; one exceedance is allowed but must be less than 50 %. Any other deviations must be described in the project narrative. The injector system must be kept very clean to meet these criteria. This requirement is applicable only to Connecticut samples.

8.3.3 Mass DEP also requires a mass discrimination check. The ratio of C_{28}/C_{20} must be > 0.85.

8.3.4 **Log Book**: All samples set up on the instrument must be entered into the run logbook (Attachment B) prior to sample analysis. The logbook must be filled out completely with the date, vial number (slot number), computer file number, method number, ESS lab ID, and the initials of the analyst setting up the instrument.

8.3.4.1 Date includes the day, month, and year.

8.3.4.2 Vial number: This field ***must*** be filled in for each entry.

8.3.4.3 Computer file ID is an instrument abbreviation, using a perpetual numbering system beginning at the start of a new year with e.g.G2F0001 and going to the end of the year.

8.3.4.4 The ESS Lab ID includes the ID of the standards, samples and all QC samples.

8.3.4.5 Initials are signed by the analyst setting up the instrument.

8.3.4.6 The Comment section is a summary of calibration results, dilution information, and any unusual observations. (Examples would include: carry over information into the sample, retention time shift, calibration standard is less than 20% recovery, sample needs dilution.)

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8.3.4.7 The Method section is the method in the chromatographic software used to operate the instrument.

8.3.5 Quality control samples are to be analyzed with their associated batches. It is not acceptable to batch QC samples together. The following is the recommended analytical sequence for an analytical batch.

8.3.5.1 Preparation Blank

8.3.5.2 Blank spike /blank spike duplicate

8.3.5.3 Environmental samples 1 to 20

8.3.5.4 Continuing Calibration verification standard (CCV)

8.3.6 A CCV is analyzed at the beginning, after every 20 samples and/or at the end of the analytical sequence. Also, the CCV must be analyzed every 12 hours. **Note: When analyzing samples for DoD/Navy/AFCEE work CCVs must be analyzed after every 10 field samples.** The final CCV must be at a different concentration than the initial CCV; alternate between 100 and 50 ppm.

8.3.7 Quantitative analysis of the Total petroleum hydrocarbons in the sample is accomplished by comparing the total area between the retention time of n-C_9 and *n*-C_{36}. The absolute retention times used to measure this range are determined from the continuing calibration standard analyzed just prior to the samples. This range is integrated with a forced baseline using the GC EnviroQuant Software.

8.3.8 The surrogate peak is also integrated.

8.4 Retention Time Windows:

8.4.1 Total Petroleum Hydrocarbon Ranges: TPH retention time (Rt) windows are defined before the Rt of the beginning marker compound (C_9) and after the Rt of the ending marker compound (C_{36}). The retention time windows are set daily by the Rt of the beginning and ending markers in the initial continuing calibration verification standard (CCV).

9.0 CALCULATIONS

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9.1 Calculation – Waters

$$mg/L = \frac{(A)(DF)(Vt)}{(CF_{avg})(Vi)(V)}$$

9.2 Calculation - Soils

$$\text{mg/Kg dry wt.} = \frac{(A)(DF)(Vt)}{(CF_{avg})(Vi)(Wt)(\%\ Solid)}$$

Where:

A = (Total Area)-(Area of Surrogate)
DF = Dilution Factor
Vt = Final Volume of Extract (ul)
Vi = Volume injected (ul)
CF_{avg} = Average of Range Calibration Factor (from Initial Calibration)
V = Volume of sample (ml)
Wt = Wet Weight of Sample
% Solid = % Solid expressed as a decimal

10.0 Quality Assurance/Quality Control Requirements

10.1 Immediately after the initial calibration, a second source standard (ICV) is analyzed. This standard is prepared at the Level 3 concentration. The percent recovery of the second source must be ±20%. If the percent recovery is outside criterion, then corrective action must be taken. Perform instrument maintenance and/or prepare a new second source standard. If the second analysis of the ICV is not within criterion, then a new calibration curve must be generated. ***Sample analysis cannot begin until a valid second source has been analyzed.***

10.2 Accuracy and Precision: All laboratory personnel must demonstrate initial proficiency for each sample preparation method/matrix that he or she performs. All new employees must successfully demonstrate initial proficiency prior to independently performing analysis on real samples. This must be accomplished by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The initial proficiency results will become part of each employee's training file.

QC Sample Preparation:

Spiking Solution: Four QC samples must be prepared from a spiking solution with the analytes of interest. The spiking solution must be made using standards **prepared independently from those used for calibration**. The QC samples must be prepared at a concentration that would result in data falling within the middle of the calibration curve. In most cases the blank spike or matrix spike solution is used. Prep: The samples are prepared in a clean matrix. In most cases this initial demonstration will simply be a matter of preparing four blank spikes with a batch of samples.

QC Sample Analysis: The four QC samples must be analyzed within the criteria of the method being evaluated. The QC samples must be handled in exactly the same manner as actual samples.

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Accuracy Calculation: Accuracy is defined as the closeness of agreement between an observed value and an accepted reference value. Each of the four spiked samples will be calculated for percent recovery. The average of the percent recovery values is the accuracy result.

Precision Calculation: Precision is defined as the agreement of a set of replicate measurements without assumption of knowledge of the true value. Precision is estimated by the relative standard deviation (RSD) of the four QC samples.

%RSD = (s / x) 100 %

Where:
s = Standard Deviation of a finite number of values. On a scientific calculator use the **σxn-1** key.

$\bar{x}$ = The average of the four QC sample % recoveries.

Reporting Accuracy and Precision**: Report Accuracy and Precision data with the following minimum info:**

Matrix: Prep Method:
Clean-up Method: Analysis Method:
Date Extracted: Date Analyzed:
Sample Prepared by: If Applicable Sample Analyzed by:

Parameter	*% Rec. QC 1*	*% Rec. QC 2*	*% Rec. QC 3*	*% Rec. QC 4*	*Average Recovery*	*Standard Deviation*	*%RSD*

10.2.1 **Interpretation of Results**: The percent recoveries should be between 50-130% and the %RSD should be less than 30%. If any of the accuracy and precision results do not fall within the criteria then re-prep and re-analyze all QC samples only for those analytes that were not within criteria.

10.3 Each day the analysis is performed the initial calibration must be verified. See Tables 1 and 2. Continuing calibration verification (CCV) must be performed at the beginning and end of each analytical sequence; see 8.2.7 for frequency of CCV. The response factors for the calibration should be within 20 percent of the initial calibration. **(The CCV concentration, when performing DoD/Navy work must be at different concentrations throughout the analytical run. One standard must be at or below the mid-range standard.)** When a CCV is out of this acceptance window, the laboratory should stop analyses and take the following corrective action:

10.3.1 If criterion is exceeded then remake and re-analyze CCV. If second consecutive CCV is within criteria then calibration is verified, otherwise re-calibrate system and re-analyze any sample analyzed after invalid CCV.

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10.3.2 If the second consecutive CCV was outside criterion, then the analyst may choose to demonstrate acceptability of initial calibration by the analysis of two consecutive CCVs at two concentrations. ***Exception: If CCV is exhibiting high bias (concentration is higher than upper limit) then any samples that are non-detect for that analyte may be reported.***

10.3.3 If the above corrective action fails, then a new five point initial calibration must be generated.

10.3.4 When performing analysis for MA MCP, Navy, USACE, DoD, or AFCEE any analyte/range outside of criteria in the CCV must be noted in the project narrative

10.4 From the beginning CCV, the retention time of the surrogate and the C_9-C_{36} range markers are determined.

Table 1

TPH (C9-C36) Criteria:						
ICAL:						
Mth	Stds	C9-C36 Rng %RSD / Linearity	Aliphatic Cmpd %RSD / Linearity	ICV	Mass Discrim.	RT Window
CT ETPH	Min of 5pts	30% / 0.99	30%/0.99	30%D	After Ical RF<20% w/ 1 <50%	Ical Avg Rt
MA TPH	Min of 5pts	25%/0.99	25%/0.99	20%D	C28/C20 >0.85	Ical Avg Rt
DOD	Min of 5pts	20%/0.995	20%/0.995	20%D	Refer to State	Ical Avg Rt
8100M	Min of 5pts	20%/0.99	20%/0.99	20%D	NA	Ical Avg Rt
CCAL:						
Mth	Stds	Mass Discrim.	C9-C36 Rng CCV	Aliphatic Analyte CCV	RT Window	
CT ETPH	Mid Pt	Beginning 12hr Seq RF<20% w/ 1 <50%	30%D End CCV recommended Every 12 hrs or 20 smp.	30%D End CCV recommended Every 12 hrs or 20 smp.	Update from beginning CCV	
MA TPH	Mid Pt	Beginning 12hr Seq C28/C20 > 0.85	25%D End CCV no failures Every 12 hrs or 20 smp.	25%D End CCV 4 failures <40% Every 12 hrs or 20 smp.	Update from beginning CCV	

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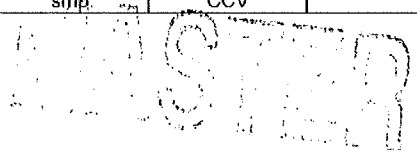
DOD	Alternate Concentration	Refer to State	20%D End CCV no failures Every 10 smp.	20%D End CCV no failures Every 10 smp.	Update from beginning CCV	
8100M	Mid Pt	NA	20%D End CCV no failures Every 12 hrs or 20 smp.	20%D End CCV 4 failures <40% Every 12 hrs or 20 smp.	Update from beginning CCV	

Table 2

DRO (C10-C28) Criteria:						
ICAL:						
Mth	**Stds**	**C10-C28 Rng %RSD / Linearity**	**Aliphatic Cmpd %RSD/Linearity**	**ICV**	**Resolution**	**RT Window**
8015	Min of 5pts	20%/0.99	20%/0.99	20%D	NA	Ical Avg Rt
ME DRO	Min of 3pts	20%/0.99	20%/0.99	20%D	C17/C18 > 60%	Ical Avg Rt
DOD	Min of 5pts	20%/0.995	20%/0.995	20%D	NA	Ical Avg Rt
CCAL:						
Mth	**Stds**	**Resolution**	**C10-C28 Rng CCV**	**Aliphatic Analyte CCV**	**RT Window**	
8015	Mid Pt	NA	20%D failures End CCV no Every 12 hrs or 20 smp.	20%D End CCV no failures Every 12 hrs or 20 smp.	Update from beginning CCV	
ME DRO	Seq Begin w/ Mid Pt & Res Std End With Mid Pt & LL Commercial Diesel	At end of Seq: C17/C18 > 60%	20%D failures End CCV no Every 20 smp.	20%D End CCV no failures Every 20 smp.	Update from beginning CCV	
DOD	Alternate Mid and Low See State Mth	NA	20%D failures End CCV no Every 10 smp.	20%D End CCV no failures Every 10 smp.	Update from beginning CCV	



10.5 **Preparation Blanks**

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10.5.1 Preparation blanks are used to verify the presence or absence of method contamination. They are prepared from controlled matrices in the same manner to that of the environmental samples.

10.5.2 Preparation blanks should follow the associated environmental samples through all phases of the sample preparation and in the analytical process.

10.5.3 For a preparation blank to be considered valid it must meet the same instrumental criteria as any other sample and the surrogate recovery of the preparation blank must meet the normal recovery criteria.

10.5.4 The preparation blank should not contain any material at a level above ½ the MRL. See section 11.0 for corrective action

10.6 **Blank Spike/Blank Spike Duplicate Samples**

10.6.1 Blank Spike/Blank Spike Duplicate Samples (BS/BSD) are prepared from a second source standard of aliphatic hydrocarbons. A 50 ppm standard is made by diluting 5 ml of the 1000 ppm standard (supplied by accustandard) to 100ml in methylene chloride. 1.0 ml of this 50 ppm solution is then spiked into a clean matrix to prepare the BS/BSD.

10.6.2 The BS/BSD must follow the associated environmental sample through all phases of the sample preparation and analysis process.

10.6.3 The BS/BSD should not contain any other material except for the spiked material at a level above the reporting limit. The BS should meet the spike recovery criteria of 40% - 140% and the % RPD between the BS and BSD must be less than 25%. In the case of the State of Connecticut, ETPH QC criteria are 60-120% for Recovery and ≤ 30% for RPD.

10.6.4 Surrogate QC recovery criteria are 40-140%, except for State of Connecticut which are 50-150 %.

10.6.5 . See Section 11.0 for corrective action.

10.7 **Matrix Spikes/Matrix Spike duplicate**

10.7.1 MS/MSDs are performed on each matrix where sample is available. The same spike is used as in 10.6.1.

10.7.2 The MS should meet the spike recovery criteria of 40% - 140% and the MS/MSD should have a %RPD of +/- 50%. In the case of the State of Connecticut, ETPH QC criteria are 50-150% for Recovery and ≤ 30% for RPD.

10.7.3 If the MS/MSD is outside of the acceptance criteria then it must be determined if it is a Matrix Effect or Laboratory error. See Section 11.0 for corrective action.

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10.8 **Method Detection Limit Study: MDLs** are determined in either reagent water or sodium sulfate and verified annually. MDLs are calculated, reported and verified according to the ESS SOP 110_0013. (Project-specific requirements may require that the MDL study be performed in the site-specific matrix.). Seven MDL samples are extracted and analyzed like any other sample in an analytical batch that contains a method blank. The MDL samples are prepared as follows:

10.8.1 **Soil MDL**: 250 ul of a 2000ug/ml Fuel Oil # 2 solution (1:10 dilution of spiking solution in 10.6.1) is added to 30g (20g for method 3541) of sodium sulfate.

10.8.2 **Aqueous MDL**: 200 ul of a 2000ug/ml Fuel Oil # 2 (1:10 dilution of spiking solution in 10.6.1) solution is added to 1 Liter of Lab DI water.

10.9 **Control charts** are generated in accordance with SOP 110.0014.

11.0 DATA VALIDATION

11.1 Data validation will be accomplished by reviewing all of the quality control parameters and assuring that they are within recommended ranges by completing the Data Review Checklist for GC/FID 8100M. The only exceptions made to ranges would be the following:

11.1.1 For MS/MSD, the RPD should be +/- 50% (≤ 30% for CT). However, there are cases where duplicates may not work. If this is the case, inform client in narrative concerning sample non-homogeneity.

11.1.2 For matrix spikes, the % Recovery should be 40-140% (50-150 % for CT). If the matrix spike is outside this range, check the BS/BSD. If the BS/BSD is within limits, matrix interferences are present and should be noted in the narrative.

11.1.3 Analytical batches with Method blanks above the ½ MRL will be re-prepped and re-analyzed with the following exceptions:

11.1.3.1 Samples that are that are at least twenty times higher than the method blank may be reported.

11.1.3.2 When the method blank is less than 5% of the regulatory limit associated with the analyte the method blank would be acceptable.

11.1.3.3 If the analyte is found in the method blank above the ½ the MRL but is not in any of the associated samples, no corrective action is needed.

11.1.3.4 Any results that are reported with method blank contamination must be B-flagged.

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11.1.4 For the BS/BSD, the % Recovery should be 40-140% (60-120 % for CT). If the BS/BSD is outside this criterion, the analytical batch will be re-extracted and re-analyzed with the following exceptions:

11.1.4.1 For BS/BSD >140% (>120% for CT) , samples with results below the MRL may be reported. It has been shown that the results above MRL would have been detected.

11.1.4.2 For BS/BSD < 40% (< 60% for CT) , samples with results above a regulatory limit may be reported.

11.1.4.3 In some instances there may be insufficient sample to re-extract. The results may be reported as estimated values when this occurs after contacting the client for instructions.

11.1.4.4 Any samples that are reported with invalid BS/BSD data must have a notation in the case narrative.

11.2 All unusual observations and method deviations will be noted in the narrative accompanying the data report presented to the client.

11.3 A second analyst reviews all data for accuracy. Results of this review are noted on the data validation checklist in the second level review column and in the comment section.

12.0 REFERENCES

12.1 SW846 Method 8015B, Nonhalogenated Organics Using GC/FID Third Edition, Update III.

12.2 SW846 Method 8100, Polynuclear Aromatic Hydrocarbons, Third Edition, Update III.

12.3 Univ. Connecticut, Analysis of Extractable Total petroleum hydrocarbons (ETPH) Using Gas Chromatograph/Flame Ionization Detection, March 1999.

12.4 NELAC Standards, Chapter 5, June 2003.

12.5 DoD QSM Final Version 4.1, April 2009.

13.0 POLLUTION PREVENTION and WASTE MANAGEMENT

13.1 ESS Laboratory's policies on pollution prevention and waste management are covered in SOP 90_0002, Hazardous Waste Contingency and Emergency Response Plan. All employees are trained in the requirements of the SOP.

14.0 METHOD PERFORMANCE

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14.1 Precision and Accuracy data must be generated by all employees before performing this analysis on client samples. The data is generated by analyzing a method blank and four blank spike samples. Acceptance criteria are 40-140% Recovery and %RSD of $\leq$ 25%.

14.2 The precision and accuracy data in Table 1 were developed using this method.

15.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

Table 1. Typical Precision and Accuracy data generated 4/23/05 – 2/14/07

Compound	Prep Method	Spk	Avg	%Rec	%RSD
Total Petroleum Hydrocarbons	3510C	20	13.38	66.9	7.87
Total Petroleum Hydrocarbons	3541	20	11.38	56.9	7.03
Total Petroleum Hydrocarbons	3550B	20	14.3	71.5	6.71

16.0 DEFINITIONS

16.1 **Accuracy**: The closeness of agreement between an observed value and an accepted reference value. When applied to a set of observed values, accuracy will be a combination of a random component and of a common systematic error (or bias) component.

16.2 **Batch**: A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 20, then each group of 20 samples or less will all be handled as a separate batch.

16.3 **Bias**: The deviation due to matrix effects of the measured value (x_s - x_u) from a known spiked amount, where x_s is the spiked sample and x_u is the un-spiked sample. Bias can be assessed by comparing a measured value to an accepted reference value in a sample of known concentration or by determining the recovery of a known amount of contaminant spiked into a sample (matrix spike).

16.4 **Control Sample**: A QC sample introduced into a process to monitor the performance of the system.

16.5 **Equipment Blank**: A sample of analyte-free media which has been used to rinse the sampling equipment. It is collected after completion of decontamination and prior to sampling. This blank is useful in documenting adequate decontamination of sampling equipment.

16.6 **Method Reporting Limit**: The lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The MRL is generally 5 to 10 times the MDL. ESS Laboratory sets the MRL to the lowest non-zero standard in the calibration curve or higher.

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16.7 **Field Duplicates**: Independent samples which are collected as close as possible to the same point in space and time. They are two separate samples taken from the same source, stored in separate containers, and analyzed independently. These duplicates are useful in documenting the precision of the sampling process.

16.8 **Blank Spike (BS)**: A known matrix spiked with compound(s) representative of the target analytes. This is used to document laboratory performance.

16.9 **Matrix**: The component or substrate (e.g., surface water, drinking water) which contains the analyte of interest.

16.10 **Matrix Duplicate**: An intra-laboratory split sample which is used to document the precision of a method in a given sample matrix.

16.11 **Matrix Spike**: An aliquot of sample spiked with a known concentration of target analyte(s). The spiking occurs prior to sample preparation and analysis. A matrix spike is used to document the bias of a method in a given sample matrix.

16.12 **Matrix Spike Duplicates**: Intra-laboratory split samples spiked with identical concentrations of target analyte(s). The spiking occurs prior to sample preparation and analysis. They are used to document the precision and bias of a method in a given sample matrix.

16.13 **Method Blank**: An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank should be carried through the complete sample preparation and analytical procedure. The method blank is used to document contamination resulting from the analytical process.

16.14 **Method Detection Limit (MDL)**: The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix type containing the analyte. See SOP 110_0013 for further explanation.

16.15 **Records**: Include all logbooks, papers, machine readable materials, or other documentary materials, regardless of physical form or characteristics.

16.16 **Surrogate**: An organic compound which is similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which is not normally found in environmental samples.

17.0 PERSONNEL QUALIFICATIONS

17.1 Analysts who perform this analysis must have a working knowledge of quantitative and qualitative analysis, instrumental methods of analysis, chemical laboratory methods, and equipment.

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17.2 All analysts, before performing any analysis, participate in the ESS Laboratory training program (SOP80_0016). The training process consists of reading the Standard Operating Procedure, gaining instruction on the procedure from an experienced analyst, and performing the initial demonstration of capability.

18.0 TROUBLESHOOTING

18.1 The following procedure is performed when the instrument is initially set up or when a continuing calibration has failed the QC criteria.

18.1.1 Set the GC system to room temperature and turn off oven.

18.1.2 Remove column by unscrewing the column in the injection port.

18.1.3 Remove septum nut and septa. *Discard septa.*

18.1.4 Remove weldment. This will expose the O-ring and glass liner. Using a set of tweezers, remove O-ring and liner. If O-ring is not distorted then set aside for later use, otherwise, replace O-ring. Remove the glass liner. In a ventilation hood rinse the liner with methylene chloride and scrub with a cotton swab. If the liner is visibly stained, then replace with a new one.

18.1.5 With a cotton swab dipped in methanol, clean the injection port and weldment.

18.1.6 Remove the gold seal nut located on the bottom of the injection port. With a cotton swab and methanol, clean the gold seal.

18.1.7 Replace all parts in the following order:

18.1.7.1 Gold seal nut. Hand tighten and ¼ turn with wrench.

18.1.7.2 Insert clean or new glass liner.

18.1.7.3 Place O-ring over liner. Slide O-ring over and down the liner until it fits snug against the injection port.

18.1.7.4 Replace weldment.

18.1.7.5 Place new green septa into weldment.

18.1.7.6 Replace septum nut. **Only hand tighten!**

18.1.7.7 Slide column nut and a new graphite ferrule over column.

18.1.7.8 Using a ceramic tile, cut 3-6 inches of the column. The cut must be square with no jagged edges.

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18.1.7.9 Connect column to injection port by inserting 3 mm of column into the injection port and hand tighten column nut. Next add ¼ turn with a wrench.

18.1.8 Make sure all gases are flowing. (Measure flows with bubble meter.) The flow should be approximately 1 ml/min going through a capillary column (0.25 um ID).

18.1.9 Turn on injection port temperature.

18.1.10Set oven temperature to 150°C and allow the system to stabilize. Bake out the oven at 300°C for an hour. Reset to 80°C.

18.2 Record all maintenance in the instrument's maintenance logbook.

19.0 Data Management And Records

19.1 **Data Management** - ESS Laboratory's utilizes the Promium Element LIMS system as part of its Data Management system. Client sample information is entered into ELEMENT LIMS and analyses are assigned to each sample. The LIMS allows EPA hold times, minimum batch QC requirements, and QC criteria to be assigned to each analysis. Standards can be entered and assigned to QC samples through the LIMS. Once analysis has been performed, data is imported using DataTool avoiding manual errors. In conjunction with Crystal Reports, the ELEMENT system allows for a wide variety of reporting formats.

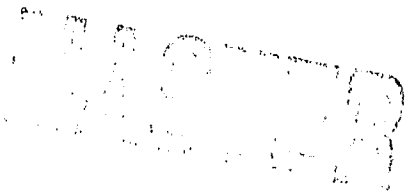
19.2 **Records** – The specific retention periods required in the NELAC Standards, EPA-CFR and state and local statutes are followed or exceeded. At a minimum, data records are retained for five years from last use (10 years for drinking water). If there is a question about whether a record should be retained or disposed because no specific requirement could be found, the record is retained until such time as a retention period is specified. Records are stored in specified-labeled locations and are easily retrievable. All raw data associated with testing is also retained including; computer printouts, chromatograms, review forms, and logbooks.

ATTACHMENTS

Attachment A – GC2 Rear Sequence Log

Attachment B – GC2 Front Sequence Log

Attachment C– Instrument Control Parameters



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Attachment A

Data Reporting **Instrument:**_____ **Matrix:**_____

ESS Laboratory (SVOA)
GC2 Rear Sequence Log

Work Orders Reported:_____

Date Analyzed: _____ Analyst: _____

	Method	Seq ID	Calibration ID
Soil			
Aqueous			

Vial	File	Lab Id	Rw	Rpt	Imp	STD ID/ Comments	Batch ID	Matrix	-RE

ICAL/CCAL Failures: _____

Comments: _____

ICAL Batch ID: BB71630(Aq); BB71631(Soil)

Final Data Review: _____
Signed copy included with each work

Control #60.0046-0801A

Lobgook Review: _____

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Attachment B

ESS Laboratory (SVOA)
GC2 Rear Sequence Log

Work Orders Reported: _____

Date Analyzed: _____ Analyst: _____

	Method	Seq ID	Calibration ID
Soil			
Aqueous			

Vial	File	Lab Id	Rw	Rpt	Imp	STD ID/ Comments	Batch ID	Matrix

ICAL/CCAL Failures: _____

Comments: _____

ICAL Batch ID: BB71630(Aq); BB71631(Soil)

Final Data Review: _____
Signed copy included with each v

Control #60.0046-0801A

Logbook Review: _____

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TOPLEVEL PARAMETERS

Method Information For: C:\HPCHEM\1\METHODS\8100SIG1.M

8100m method as of 5.15.01

Method Sections To Run:

( ) Save Copy of Method With Data
( ) Pre-Run Cmd/Macro =
(X) Data Acquisition
(X) Data Analysis
( ) Post-Run Cmd/Macro =

Method Comments:
8100m

END OF TOPLEVEL PARAMETERS

INSTRUMENT CONTROL PARAMETERS

Sample Inlet: GC
Injection Source: GC ALS

HP GC Injector

Front Injector:

Sample Washes	2
Sample Pumps	2
Injection Volume	1.0 microliters
Syringe Size	10.0 microliters
On Column	Off
Nanoliter Adapter	Off
PostInj Solvent A Washes	2
PostInj Solvent B Washes	2
Viscosity Delay	2 seconds
Plunger Speed	Fast

Back Injector:
No parameters specified

HP5890 Temperature Parameters

Zone Temperatures:	State	Setpoint
Inlet A:	On	300 C
Inlet B:	On	300 C
Detector A:	On	315 C
Detector B:	On	315 C

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Oven Parameters:

Oven Equib Time:	0.20 minutes
Oven Max:	320 C
Oven State:	On
Cryo State:	Off
Cryo Blast:	Off
Ambient:	25 C

Oven Program:

Initial Temperature:	80 C
Initial Time:	2.00 minutes

Level	Rate (C/minute)	Final Temperature (C)	Final Time (minutes)
1	20.0	265	0.00
2(A)	30.0	320	12.00
3(B)	0.0	0	0.00

Next Run Time: 25.08 minutes

HP5890 Purge Valve Settings

Inlet Purge	Init Value	On Time	Off Time	Splitless Injection
A	On	0.75	0.00	Yes
B	On	0.75	0.00	Yes

HP5890 Valve and Relay Information

Initial Setpoints:

5890 Valves:

Valve 1: Off Valve 2: Off Valve 3: On Valve 4: On

19405 Valves:

Valve 5: Off Valve 6: Off Valve 7: Off Valve 8: Off

19405 Relays:

Relay 1: Off Relay 2: Off Relay 3: Off Relay 4: Off

HP5890 Detector Information

Detector	Type	State
A	FID	On
B	FID	On

HP5890 Signal Information

Save data for signal 1 only.

Signal	Source	Peak Width	Data Rate	Start Data	Stop Data
	Det A	0.053	5.000	2.20	27.00
	Det B	0.053	5.000	2.50	20.00

END OF INSTRUMENT CONTROL PARAMETERS

DATA ANALYSIS PARAMETERS

Method Name: C:\HPCHEM\1\METHODS\8100SIG1.M

Percent Report Settings

Sort By: Signal

Output Destination
Screen: No
Printer: Yes
File: No

Integration Events: AutoIntegrate

Ge ate Report During Run Method: No

Signal Correlation Window: 0.020

Qualitative Report Settings

Peak Location of Unknown: Apex

Library to Search	Minimum Quality
DEMO.L	0

Integration Events: AutoIntegrate

Report Type: Summary

Output Destination
Screen: No
Printer: Yes
File: No

Generate Report During Run Method: No

Quantitative Report Settings

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Report Type: Summary

Output Destination
Screen: No
Printer: Yes
File: No

Generate Report During Run Method: Yes

Alkanes-front-GC4
Calibration Last Updated: Wed May 09 10:39:32 2001

Reference Window: 10.00 Percent
Non-Reference Window: 5.00 Percent
Correlation Window: 0.02 minutes
Default Multiplier: 1.00
Default Sample Concentration: 0.00

Compound Information

---

1) O-Terphenyl ( )

Ret. Time 11.28 min., Extract & Integrate from 11.18 to 11.38 min.

Sig	1	Rel Resp.	Pct. Unc.(rel)	Integration
Tgt	TIC			*** AUTO ***

Lvl	ID	Conc (ppm)	Response
cc		50.000	740010
50		50.000	740010
10		10.000	140447
20		20.000	304691
100		100.000	1495970
250		250.000	4550776
500		500.000	8920253

Qualifier Peak Analysis OFF
Curve Fit: Avg. RF

---

2) C9-C36 ( )

Ret. Time 12.95 min., Extract & Integrate from 3.20 to 22.70 min.

Signal		Rel Resp.	Pct. Unc.(rel)	Integration
Tgt	TIC			*** AUTO ***

Lvl	ID	Conc ( )	Response
cc		700.000	14024122
50		700.000	14024122
10		140.000	2790277
20		280.000	4246069

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100	1400.000	21737878
250	3500.000	58809718
500	7000.000	111385989

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

END OF DATA ANALYSIS PARAMETERS

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ESS Laboratory
Division of Thielsch Engineering
Cranston, RI

SOP NO. 60_8270
SEMIVOLATILE ORGANIC COMPOUNDS ANALYSIS BY GAS CHROMATOGRAPHY/ MASS SPECTROMETRY
(EPA Method 625/SW-846 METHOD 8270C and D)

APPROVED BY:

Operations Manager	Date 8/17/10
QA Manager	Date 8/17/10
Laboratory Director	Date 8/17/10

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SEMIVOLATILE ORGANIC COMPOUNDS ANALYSIS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (EPA Method 625/SW 846 METHOD 8270C and D)

1.0 SCOPE AND APPLICATION

1.1 To determine the concentration of semi-volatile organic compounds in extracts prepared from all types of solid waste matrices, soils, and waters. Parameters which may be measured are as follows:

Analyte	Analyte
1,4-Dichlorobenzene-d4	Dimethylphthalate
N-Nitrosodimethylamine	Acenaphthylene
Pyridine	2,6-Dinitrotoluene
2-Fluorophenol	2-Nitroaniline
bis(2-Chloroethyl)ether	Acenaphthene
Phenol-d5	2,4-Dinitrophenol
2-Chlorophenol	Dibenzofuran
Phenol	4-Nitrophenol
Aniline	3-Nitroaniline
2-Chlorophenol-d4(SURR)	2,4-Dinitrotoluene
1,3-Dichlorobenzene	Fluorene
1,4-Dichlorobenzene	2,3,4,6-Tetrachlorophenol
1,2 Dichlorobenzene-d4	Diethylphthalate
1,2-Dichlorobenzene	4-Chloro-phenyl-phenyl ether
Benzyl Alcohol	Phenanthrene-d10
bis(2-chloroisopropyl)Ether	4-Nitroaniline
2-Methylphenol	4,6-Dinitro-2-Methylphenol
Acetophenone	N-nitrosodiphenylamine
N-Nitroso-Di-n-Propylamine	Azobenzene
Hexachloroethane	2,4,6-Tribromophenol
3+4-Methylphenol	4-Bromophenyl-phenylether
Naphthalene-d8	Hexachlorobenzene
Nitrobenzene-d5	Pentachlorophenol
Nitrobenzene	Phenanthrene
Isophorone	Anthracene
2-Nitrophenol	Carbazole
Benzoic Acid	Di-n-butylphthalate
2,4-Dimethylphenol	Fluoranthene
bis(2-Chloroethoxy)methane	Benzidine
2,4-Dichlorophenol	Chrysene-d12
1,2,4-Trichlorobenzene	Pyrene
Naphthalene	Terphenyl-d14
4-Chloroaniline	Butylbenzylphthalate
Hexachlorobutadiene	3,3'-Dichlorobenzidine

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4-Chloro-3-Methylphenol	Benzo(a)anthracene
2-Methylnaphthalene	Chrysene
1-Methylnaphthalene	bis(2-Ethylhexyl)phthalate
Acenaphthene-d10	Perylene-d12
Hexachlorocyclopentadiene	Di-n-octylphthalate
2,4,6-Trichlorophenol	Benzo(b)fluoranthene
2,4,5-Trichlorophenol	Benzo(k)fluoranthene
2-Fluorobiphenyl	Benzo(a)pyrene
1,1'-Biphenyl	Indeno(1,2,3-Cd)Pyrene
2-Chloronaphthalene	Dibenzo(a,h)Anthracene
2,2'-Oxybis(1-chloropropane)	Benzo(g,h,i)perylene
1,2,4,5-Tetrachlorobenzene	Pentachloronitrobenzene
Caprolactam	

IS = Internal standard **Surr** = Surrogate

1.2 This method can be used to quantitate most neutral, acidic and basic organic compounds that are soluble in methylene chloride and capable of being eluted without derivatization as sharp peaks from gas chromatographic fused silica column coated with slightly polar silicone.

1.3 Some compounds may require special treatment when being determined by this method. (See Interference and Potential Problems, section 5.0)

2.0 METHOD SUMMARY

2.1 Prior to using this method, samples are prepared for chromatography using the appropriate preparation and cleanup methods. A measured volume or weight of sample (approximately 1 L for liquids, 2 g to 30 g for solids) is extracted using the appropriate sample extraction technique. Liquid samples are extracted at acidic pH with methylene chloride using a continuous liquid-liquid extraction (SOP 50_3520C). Solid samples are extracted with methylene chloride using either manual or automated Soxhlet extraction (SOP 50_3540B or 50_3541), or Microwave Extraction (SOP 50_3546) methods. A variety of cleanup steps may be applied to the extract, depending on (1) the nature of the co-extracted matrix interferences and (2) the target analytes. After cleanup, the extract is analyzed by injecting a 1-uL sample into a gas chromatograph with a narrow-bore fused silica capillary column and mass spectrometer detector (GC/MS). This method describes the appropriate chromatographic conditions to achieve separation for a qualitative and quantitative GC/MS analysis.

3.0 HEALTH AND SAFETY

3.1 Each employee has been trained and has acknowledged being trained in the safe use and handling of chemicals being used in the laboratory. This training has been performed according to the ESS Training SOP 80_0016 and by the Chemical Hygiene Plan, SOP No. 90_ 001, in conjunction with the Safety orientation.

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3.2 All sample and material handling should be done in a hood while using proper protective equipment to minimize exposure to liquid or vapor. Minimum personnel protective equipment includes the use of laboratory safety glasses, a lab coat or apron, and protective gloves.

3.3 The MSDS for the concentrated chemicals used in the area are kept on file in a central location that is available for all employees to review.

3.4 Several chemicals used in this procedure are tentatively classified as known or suspected carcinogens. Extreme caution should be used in handling all chemicals in this procedure.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

4.1 Aqueous samples are collected in 1 Liter amber borosilicate glass jars with Teflon lined caps. The samples are stored in a dark walk-in cooler at 4°C. Two liters should be provided so samples can be re-extracted when necessary. Aqueous samples must be extracted within 7 days from date sampled.

4.2 Soil/sediment samples are collected in 4 – 8 ounce jars with Teflon lined caps. The samples are stored in a dark walk-in cooler at 4°C. Up to thirty grams of sample are required for extraction and ten grams is required to determine the percent solids. One hundred grams should be provided so samples can be re-extracted when necessary. Soil/Sediment samples must be extracted within 14 days of date sampled.

4.3 All extracts are stored in 2 ml Teflon capped vials in the extract storage refrigerator located in the SVOA lab. These extracts are stored at 4°C and must be analyzed within 40 days of date extracted.

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

5.1 Blanks, samples and spikes must be evaluated for interferences. The source of interference should be identified and corrective action should be taken to eliminate the problem.

5.2 Contamination by carry over can occur whenever high concentration and low concentration samples are sequentially analyzed.

5.3 Contamination by phthalate esters can occur during sample preparation. Common flexible plastics contain varying amounts of phthalate esters that are easily extracted or leached from such materials during laboratory operations. Cross-contamination of clean glassware routinely occurs when plastics are handled. Avoid contact with any plastic materials and check all solvents and reagents for phthalate contamination to minimize interferences from phthalate esters. Exhaustive cleanup of solvents, reagents and glassware may be required to eliminate background phthalate ester contamination.

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5.4 Benzidine may be subject to oxidative losses during solvent concentration and its chromatographic behavior is poor. Estimated values are reported at 50ug/L for a 1 liter water extraction and 1650ug/Kg for a 30 gram soil extraction.

5.5 Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition.

5.6 N-nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be separated from diphenylamine.

5.7 Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, benzoic acid, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.

5.8 Since 1,2-Diphenylhydrazine oxidizes to Azobenzene in the GC injection port, it is indistinguishable from Azobenzene on the GC/MS and will be reported as Azobenzene.

6.0 EQUIPMENT/APPARATUS

6.1 Gas Chromatography/Mass Spectrometer system

6.1.1 **Gas Chromatography**: HP 5890 series II with EPC (electronic pressure control)

6.1.2 **Column**: J&W DB-5MS, HP-5MS or RESTEK RXI-5MS (30m X 0.25 mm ID with 25 um film thickness) or equivalent.

6.1.3 **Mass Spectrometer**: HP 5971, HP 5972 or equivalent, capable of scanning from 35-500 amu every 1 second or less, using 70 volts (nominal) electron energy in the EI mode. The MS must be capable of meeting the criteria in Table 2 when 50 ng of DFTPP (Decafluorotriphenylphosphine) is injected.

6.1.4 **GC/MS interface**: Must give an acceptable response at a 50-ng injection and Pass the tuning criteria on Table 2.

6.2 Data System: Data system:

6.2.1 **Computers:** The Semi-Volatiles laboratory has three GC/MS systems analyzing method 625/8270C. SVOA MS1 has an AST computer with a Windows 95 operating system. SVOA MS2 has a DELL computer with a Windows NT operating system. SVOA MS4 has MCP computer with Windows XP operating system. All computer systems are networked to a Windows 2000-server, which is the destination of all files. A differential back-up is performed nightly and a full back is performed each weekend using Veritas Backup Exec to tapes. As the

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systems acquire and store data onto the server, the server becomes full. The data is downloaded and archived onto CDs.

6.2.2 **Software:** HP/Agilent Environmental Chemstation and Shimadzu GCMS Solution, Version 2.50 SV3 (MS4) - The software is interfaced to the mass spectrometer detectors and allows the continuous acquisition and storage on machine-readable media of all chromatograms obtained throughout the duration of the instrument program. The software is capable of integrating the abundance in any EICP between specified times. Current Chemstation versions SVOA MS1: G1032C version C.01.00 and SVOA MS2: G1701BA Version B.01.00.

6.3 10µL, 25µL, 100µL, 500µL, 1000µL **Hamilton syringes**.

6.4 **Volumetric Flasks**: Class A

6.5 **Balance**: Top loading, OHAUS,® Precision Standard

6.6 **Sample vials**: Glass with Teflon-lined crimp tops

7.0 REAGENTS AND STANDARDS

7.1 **Reagents**: Reagent grade or better chemicals shall be used in all tests. If the purity of a reagent is in question, it is analyzed for contamination. If the target analytes concentration is less than the ½ MRL in the method blank, then the reagent is acceptable.

7.1.1 Organic free reagent water. (ESS DI water system)

7.1.2 Methylene Chloride, HPLC pesticide grade.

7.1.3 Acetone, HPLC pesticide grade.

7.2 **Standards**:

7.2.1 All Primary, Stock, and working standard information is tracked in logbooks according to SOP 60_0001 and also in the Promium Element LIMS.

7.2.2 **Primary Standards**: The certificates of analysis in Attachment A detail the individual analytes and concentrations in each of these standard mixes. The primary standards are purchased as certified mixes and stored in a dark refrigerator at 4 ± 2°C. Primary standards, whether opened or unopened, are not used after the manufacturer's expiration date or eighteen months, whichever comes first. The refrigerator is located in the SVOA lab. Standards must not be stored with samples.

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Standard	Source	Vendor	Catalog No.	Conc. (µg/ml)
8270 Mix #1	Primary	Restek	31850	500-1000
8270 Mix #2	Primary	Restek	31806	1000
8270 Mix #3	Primary	Restek	31825	2000
8270 Mix #4	Primary	Restek	31879	2000
SemiVOA Mix 1	Secondary	Ultra	SVM-8270	1000
SemiVOA Mix 1	Secondary	Ultra	SVM-8271	1000
Custom Standard Mix 3	Secondary	Ultra	CUS-7266	2000
Custom Standard Mix 4	Secondary	Ultra	CUS-7270	2000
Benzidines Mix	Secondary	Ultra	US-105N	2000
Acid Surrogate	-	Restek	31083-510	7500
Base/Neutral Surrogate	-	Restek	31082-510	5000
SV Internal Std Mix	-	Restek	31006	4000

7.2.3 **Primary Calibration Stock Standard** The stock standard is used to prepare the calibration standards. It is stored in a 15 ml Teflon lined cap in a refrigerator at 4°C. The stock standard must be replaced after twelve months, or sooner if comparisons with quality control check samples indicate a problem. If any of the primary standards used to prepare the stock standard have expired the stock standard must be discarded. All of the primary standards used to prepare the stock standard should be taken out of the refrigerator and allowed to come to room temperature prior to use - sonicate standard per manufacturer's instructions. Into a 10 ml volumetric flask transfer 2.0 ml each of Mixes 1 & 2 and 1 ml each of Mixes 3 & 4 (7.2.2) together with 267 µL of Acid Surrogate and 400 µL of Base/Neutral surrogate. Volumize to 10.0 ml with Methylene chloride. The volumes of the standards are measured with a 1.0ml Hamilton syringe. This results in a stock solution containing all of the target analytes and surrogates at a concentration of 200 µg/ml. ***Expires in 12 months***.

7.2.4 **Second Source Stock Standard**: The second source stock standard is prepared from the five certified standards from Ultra Scientific (see 7.2.2). 2.0 ml each of Mixes 1 & 2 and 1 ml each of Mixes 3, 4 & 5, & 267 µL of Acid Surrogate and 400 µL of Base/Neutral surrogate is transferred to a 10-ml Class A volumetric and brought to volume with Methylene chloride. The concentration of the SVOA analytes in this solution will be 200 µg/ml. This solution is transferred to a 15-ml vial. The vial is labeled Second Source Semi-VOA Stock Standard with the date prepared, expiration date, concentration, preparer's initials, and the ESS working standard ID number. The standard is stored in the SVOA standard refrigerator. ***Expires in 12 months***.

7.2.5 **Working Standards**:

7.2.5.1 **Tuning Standard**: Ready to use as purchased after transfer to a 1ml vial. Purchased from Supelco, Catalog # 4-7387 at 50 µg/ml or Absolute Standards Cat # 43030.

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7.2.5.2 **Calibration Standards**: Eight calibration standards are prepared from the Primary stock standard (7.2.3). All standards are diluted to volume with Methylene chloride. Standards are stored at 4°C and prepared every six months, or sooner if standards present a problem. One ml of each calibration level is prepared separately in a 2ml vial. Prior to analysis internal standard is added to each of the calibration levels. Using a 25µL syringe, 10µL of the internal standard from Restek (See 7.2.2) is injected into each of the 1.0 ml calibration standards.

Level No.	Standard ID	Volume of Stock	Final Volume	Analyte Conc.	Internal Std. Conc.
1	STD005	25 µL	1.0 ml	5 µg/ml	40 µg/ml
2	STD010	50 µL	1.0 ml	10 µg/ml	40 µg/ml
3	STD025	125 µL	1.0 ml	25 µg/ml	40 µg/ml
4	STD050	250 µL	1.0 ml	50 µg/ml	40 µg/ml
5	STD080	400 µL	1.0 ml	80 µg/ml	40 µg/ml
6	STD120	600 µL	1.0 ml	120 µg/ml	40 µg/ml
7	STD160	800 µL	1.0 ml	160 µg/ml	40 µg/ml
8	STD200	1000 µL	1.0 ml	200 µg/ml	40 µg/ml

7.2.5.3 **Second Source Working Standard**: 250 µL of the second source stock standard (7.2.4) is measured with a 1000µl syringe and diluted to 1000µL in the syringe with Methylene chloride. The final concentration of the analytes in this solution will be 50µg/ml. The solution is transferred to a 2ml-target vial and capped. Using a 25 µL syringe, 10µL of the internal standard from Restek (7.2.2) is injected into the 1.0 ml second source standard. The target vial is labeled as Second Source STD with the working standard ESS lab ID. This standard expires six months after date prepared, or sooner if standards present a problem.

7.2.5.4 **Semi-volatile Surrogate Solution**: This solution contains all acid and base/neutral surrogates. One ml of this solution is spiked into each blank, BS, BSD, sample, MS and MSD ,(for extracts with a final volume of 1ml.). One-half ml (0.5) of this solution is spiked into each blank, BS, BSD, sample, MS and MSD (for extracts with a final volume of 0.5ml.).Prepare by obtaining a 5 ml ampoule each of the acid surrogate Base/Neutral surrogate in section 7.2.1. Use a 5 ml class A pipette to transfer 5 ml of each of the ampoules into a 250 ml volumetric. Dilute the surrogate to the 250 ml mark with Acetone. Cap and mix the solution by inverting the volumetric 3 to 5 times. Transfer the solution into a 250 ml amber glass bottle. The bottle is labeled with the working standard ID, name of the solution, concentration, preparer's initials, and expiration date. The expiration date is either the expiration date of the primary standard or six months from date prepared whichever comes first. Before this solution can be used, the concentration of the target

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analytes must be verified. The laboratory will analyze 1.0 ml of this solution like a sample. The result must fall within 15% of the true value.

7.2.5.5 **Semi-volatile Spike Solution**: 5.0 ml each of Mixes 1 & 2 and 2.5 ml each of Mixes 3, 4 & 5 are added to a 50 ml volumetric flask **(section 7.2.2)**. The solution is brought to volume with Acetone. Before the organic prep laboratory can use the spike, the spiking solution is analyzed by taking 1 ml and adding 10 µL internal standard. The percent recoveries are then calculated. If the recoveries are between 80% and 120%, then the lab can use the spiking solution. If not, then a new batch of standard should be used to prepare more of the spiking solution.

8.0 PROCEDURE

8.1 ESS Laboratory's policy is that the audit trail on the Chemstation/Enviroquant software is always on. This ensures that any changes made to the instrument operating method be documented through the audit trail.

8.2 Sample Preparation: Samples must be prepared by one of the following methods:

Matrix	Method	Matrix Summary
Water	3510C	Separatory Funnel (PAH only)
Water	3520C	Liquid-Liquid Extraction
Soil	3540	Manual Soxhlet Extraction
Soil	3541	Automated Soxhlet Extraction
Soil	3546	Microwave Extraction

8.3 **Instrument Setup**:

8.3.1 **GC/MS operating conditions**: A detailed description of the instrument operating parameters is in the Enviroquant GC/MS method in Attachment B.

Mass Range	35-500 amu
Scan time	< 1 sec/scan
Initial Temp	40, hold for 2 min
Temp program 1	40-130 at 35/min
Final Temp 1	130, hold for 0.00 min
Temp program 2	130-300 at 12/min
Final temp 2	300, hold for 13 min or until Benzo[g,h,i]perylene elutes
Inj Temp	250 - 275
Transfer line temp	300
Source temp	Manufacturer's specs
Injector	HP EPP (Pulsed pressure program)
Sample volume	1 µl
Carrier Gas	Helium at 1.5 ml/min.

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8.3.2 **Mass Spectrometer Evaluation**: Prior to placing an MS into service and after major maintenance, its performance must be evaluated.

8.3.2.1 **Auto Tune**: The instrument is first tuned using the standard Spectra Auto tune from the top menu. See Chapter 3 – "To Interpret the Tune Report" in HP G1034C MS ChemStation User's Guide. This report is printed and placed in the maintenance run log.

8.3.2.2 **Instrument Tune** using HP Software: From the top menu, "Tune MS"⇒"Target Tune"⇒"Tune"⇒"DFTPP Tune". This tunes the MS to preprogrammed targets: Mass 131 32%, Mass 219 32%, and Mass 50 2% with a target peak width of 0.45 amu. The resulting spectra should be close to this target.

8.3.2.3 "Tune Ms" ⇒ "Manual Tune" ⇒ "Load DFTPP.U" ⇒ "Adjparam" can also be used to set the target ions. Check for Air Mass: 28 (Nitrogen), 32 (Oxygen), 18 (Water), and 44 (Carbon Dioxide). All should be less than 10% of the Base 69 Ion. The 218 Ion should be between 30-35% and the 502 Ion should be between 1-2%.

8.3.2.4 Analyst judgment, per instrument history, determines if MS is functioning correctly. Compare prior results to current operating conditions to evaluate the performance of the MS. If the electron multiplier voltage needs to be increased drastically then the mass spec probably needs to be cleaned.

8.4 **Loading the instrument**: All standards and samples are analyzed in 2 ml target vials designed to fit the HP auto-sampler. The target vials are labeled with the ID of the standard or sample using a fine point marker. The tray on the auto-sampler is numbered 1-99. The instrument is set up with one injection system. The Tune and subsequent standards are placed in slots 1-9. This is the standard setup for the calibration standards. Vials can be placed in different slots as long as the slot number is written in the logbook.

Tray # 2	DFTPP Tune Standard
Tray # 3	SVOA Standard Level-1
Tray # 4	SVOA Standard Level-2
Tray # 5	SVOA Standard Level-3
Tray # 6	SVOA Standard Level-4
Tray # 7	SVOA Standard Level-5
Tray # 8	SVOA Standard Level-6
Tray # 9	SVOA Standard Level-7
Tray # 10	SVOA Standard Level-8
Tray # 11	SVOA Second Source Standard Level –4

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8.5 **Log Book**: All samples set up on the instrument must be entered into the run logbook. All logbook entries are performed prior to sample analysis. The logbook must be filled out completely as follows: (See Attachment F)

8.5.1 **Date**: include the day, month, and year of analysis.

8.5.2 Vial number: This is a required field.

8.5.3 **Computer file ID**: GC/MS Data is stored in a network directory (Q drive). Data is stored in the appropriate instrument directory (Q:\SVOA\MS2_mf). Each day a batch is set up, a new sub-directory is created with the instrument ID, month, day, and year (Q:\SVOA\MS2_ME\MF022802). Within this directory, data is stored in the following format: A file folder is created for each sample. The ID of each sample file folder has an instrument number followed by a sequential number.

Ex.: SV217823

Where: SV2 is SVOA GC/MS 2
17823 is the 17,823rd file.

8.5.4 **ESS Lab ID:** includes the ID of the standards, samples and all QC samples.

8.5.5 **Analyst's initials**

8.5.6 **Comments**: used for calibration standard IDs, dilution information, and any unusual observations.

8.5.7 **Method 8270/625**: The method in the chromatographic software used to operate and calibrate the instrument. For the 8270 analysis a method could have the ID SV2EA, where: "SV" stands for Semi-Volatile, "2" is the instrument number, and EA is a sequential letter sequence. A copy of a method is in Attachment B.

8.5.8 **Element** Sequence ID and Calibration ID, for soil and water sequences.

8.5.9 RW: Column to indicate samples which have been reviewed.

8.5.10 RPT: Column to indicate runs to be reported.

8.5.11 Imp By: Column to indicate data has been imported into the LIMs system.

8.6 **Initial Calibration**:

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8.6.1 A tune check is performed by analyzing a 50ng injection of DFTPP solution described in section 7.2.5.1. The mass spectra of DFTPP ***must*** meet the criteria

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in Table 2 prior to standard or sample analysis. Analysis does not begin until all criteria are met. A background subtraction is done when needed to eliminate column bleed or background ions. Do not subtract part of the DFTPP peak.

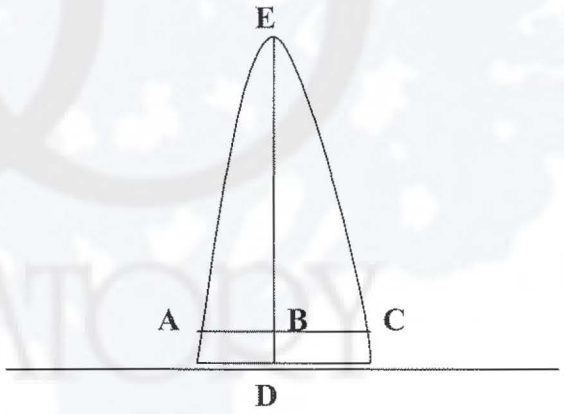
8.6.2 The GC/MS tuning standard is also used to assess GC column performance and injection port inertness. Degradation of DDT to DDE and DDD should not exceed 20%. The area count of each of these three analytes is measured. The % Breakdown is calculated as follows:

$$\% \text{ DDT Breakdown} = \frac{\text{Area DDE} + \text{Area DDD}}{\text{Area DDT} + \text{Area DDE} + \text{Area DDD}} \times 100\%$$

8.6.2.1 Benzidine and Pentachlorophenol are also present in the DFTPP mix. There should be no visible peak tailing for either of these compounds. Peak tailing is calculated with the following formula. The tailing factors for pentachlorophenol and benzidine should not be greater than 2.

Tailing = BC/AB

Peak Height = DE
10% Peak Height = BD
Peak Width at 10% Peak Height = AC
Apex = E



8.6.2.2 If degradation is excessive and/or poor chromatography is noted, the injection port may require cleaning. See daily instrument maintenance. The results of the tune are checked and recorded.

8.6.2.3 If the criteria, set in section 8.6.2.1-8.6.2.2, are not all met, a notation must be made on the data review checklist. When performing analysis for US Dept of Defense (DOD), all criteria must be met before analysis begins.

8.6.2.4 All subsequent standards, blanks, samples and spikes associated with the DFTPP analysis must use the same mass spectrometer conditions. The 12 hour tune time starts with the injection time of the DFTPP solution.

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8.6.3 10 µL of internal standard solution in section 7.2.1 is spiked into each 1 ml of the calibration standards. In most cases, the base peak ion of each internal standard is used as the primary ion for quantitation. The specific ions used for quantitation of each analyte are listed in the GC EnviroQuant method, (Attachment B). If interferences are present then the next most intense ion is used (For 1,4-Dichlorobenzene-d4 m/z 152 is used for quantitation.) The internal standard solution ID must be entered into the run log page for that run.

8.6.4 After the tune has passed, 1 µL each of the calibration standards is injected and analyzed. Response factors are then calculated for each compound as follows:

RF = (AxCis)/(AisCx)

Where:

Ax = Area of the primary ion for the compound being quantitated
Ais = Area of primary ion for the internal standard
Cis = Concentration of the internal standard (ng/µl)
Cx = Concentration of compound being measured (ng/µl)

8.6.5 The average Response Factor as well as the percent relative standard deviation (%RSD = [SD/RF] x 100) is calculated for each compound. The %RSD should be ≤ 20% for each compound. If this criterion is met, then the relative response factor is considered constant over the calibration range, and the average relative response factor may be used for quantitation. If the %RSD of any compound is greater than 20%, construct calibration curves of area ratio (A/Ais) versus concentration ratio using first or higher order regression fit of the five calibration points. A minimum of five consecutive points are needed for average response or linear regression. A minimum of six consecutive points are needed for quadratic regression. The analyst should select the regression order that introduces the least calibration error into the quantitation.

8.6.5.1 If more than 10% of the compounds in the initial calibration exceed an RSD of 20% and fail the minimum COD for linear or quadratic regression, clean or replace the injector liner and/or column, then re-calibrate. For each of the calibration compounds the relative retention times should not shift more than 0.06 relative retention time units from the mid-level calibration standard. *Note: For linear and quadratic regression curve, a minimum coefficient of determination of 0.99 is required. The Enviroquant software does not show the COD (R^2) for quadratic analysis. The analyst must enter the calibration into ELEMENT to check the COD on quadratic curves.*

8.6.5.2 A minimum response factor for the most common analytes (see Table 6 for SW846-8270D; MADEP-CAM requires RF≥0.05) should be demonstrated for each calibration level. Some compounds may fail these criteria. *If critical project compounds fail the minimum RF criteria, then*

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corrective action must be taken. Possible problems include standard mixture degradation, injection port inlet contamination, contamination at front end of column, and active sites within the chromatographic system.

NOTE: After generating the initial calibration curve in Enviroquant, the analyst must visually check that each calibration standard was entered into the new calibration method. This is accomplished by checking that the area response for one compound or range from each calibration standard's printout corresponds to the area count listed in the calibration method in Enviroquant

8.6.6 After the Initial calibration has been generated, the analyst must create a copy of the Standard 2 file and quantitate the standard with the new calibration. Review of the analyte recoveries in the standard will demonstrate the appropriateness of the calibration curve.

8.6.7 The RRT's are established from the 50ppm mid point standard in the initial calibration.

8.6.8 A second source standard is run after the initial calibration to verify the primary standards. The second source should be between 70-130% (80 – 120% for DOD) of the expected concentration, see Section 10.0 for extended criteria.

8.7 **Daily GC/MS Calibration**: (Continuing Calibration)

8.7.1 Prior to sample analysis a GC/MS tuning standard must be analyzed. A 50 ng injection of DFTPP must meet the same criteria in Section 8.6.1. This criteria must be met each 12 hour shift. See Table 2. The ID and injection time for the tune standard must be entered into the comment section of the instrument log.

8.7.2 A calibration standard at a mid-calibration range is run immediately after the DFTPP (SSTD050 at 50 ng/µl). The ID for the SSTD050 Standard must be entered into the comment section of the instrument run log. The standard is run each 12 hour shift immediately after the tuning standard. The response factor data is then compared with the average response factor data from the initial calibration. The following formula is used to calculate the percent difference:

$$\% \text{ Difference} = \frac{\overline{RF} - RF}{RF} \times 100\%$$

Where:

$\overline{RF}$ = Average Response factor from the initial calibration.
RF = Response factor from current check standard.

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8.7.2.1 Alternatively, if the initial calibration for an analyte consists of a first or higher order curve, the following equations is used to calculate the % drift:

$$\%Drift = \frac{50 - Cx}{50} \times 100$$

Where: Cx = Concentration in ng/ul of the analyte

The % Difference/Drift ***must*** be less than 20% for the initial calibration to be assumed valid and the minimum RF must be achieved. If these criteria are not met for for more than 20% of the compounds included in the ICAL, corrective action must be taken prior to analysis of samples (HOWEVER, see Appendix E for 8270C requirements and Appendix F for 8270D requirements). In cases where compounds fail, they may still be reported as non-detects if it can be demonstrated that there was adequate sensitivity to detect the compound at the applicable quantitation limit. When the failed compound is present, the concentrations must be reported as estimated values.

8.7.3 The internal standard responses and retention times are evaluated in the calibration check. If the retention times shift more than 0.5 minutes from the mid-point calibration standard in the most recent initial calibration, then the GC/MS system must be inspected for possible malfunctions. Also, if the internal standard area shifts more than a factor of two (50 to 200%) from the mid-point calibration standard in the most recent initial calibration, then the GC/MS must be inspected and appropriate corrections made. This usually involves instrument maintenance.

8.7.4 The relative retention times of each compound in each calibration run should agree within 0.06 relative retention time (RRT) units. The RRTs in the continuing calibration standard is compared to the RRTs from the 50ppm std in the ICAL.

8.7.5 A method blank should be run after the mid-range standard, or at some point in the analytical run to ensure the analytical system is free from contamination.

8.8 **Sample Analysis**:

NOTE: It is not acceptable practice to group QC samples together and/or to analyze QC samples on one instrument and their associated samples on another instrument. Analyst must try to analyze batch QC samples, as capacity allows, along with their associated field samples.

8.8.1 Just prior to sample analysis 10µL of internal standard is spiked into each 1.0 ml of sample extract. The samples are analyzed under the same conditions as the initial calibration standards. The internal standard identification must be recorded in the run log.

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8.8.2 Samples must be analyzed within the 12-hour tune time, which begins at the injection time of the tune standard. Any other analysis run after the 12-hour tune time, i.e., 625, Siloxanes and Pesticides must be identified as such.

8.8.3 Sample dilution is made when the response of any quantitation ion exceeds the initial calibration curve. Additional internal standard is always added to the diluted extract to maintain the required 40 ng/µL concentration of each internal standard. Dilutions are also made when samples will not inject into the instrument due to their viscosity.

8.9 **Qualitative analysis**:

8.9.1 **Target Compound List**: An analyte is identified by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound. The standard mass spectra are obtained from the SSTD050 (Level 3) of the initial calibration. Two criteria must be satisfied to verify identification:

8.9.1.1 The sample component must elute at the same relative retention time as the standard component. It must elute within ±0.06 RRT units of the RRT of the standard. The RRT of the sample component is compared to the RRT from the 50ppm std in the ICAL.

8.9.1.2 The sample and standard mass spectra should correspond. All ions greater than 10% in the standard mass spectrum must be present in the sample mass spectrum. The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Ex. An ion with a relative ratio of 50% in the standard must be between 20 and 80% in the sample.)

8.9.2 **Tentatively Identified Compounds**: For samples containing components not associated with the calibration standard, a library search can be made for tentative identification. Guidelines for tentative identification are as follows:

8.9.2.1 Relative intensities of all major ions greater than 10% should be present in the sample spectrum.

8.9.2.2 The relative intensity of major ions should agree within plus or minus 20%. (Ex. An ion with a relative ratio of 50% in the standard must be between 30 and 70% in the sample.)

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8.9.2.3 Molecular ions present in the reference spectra should also be in the sample spectrum.

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8.9.2.4 Ions not present in the reference spectra should be reviewed for possible background contamination. Background subtractions should be attempted to account for this.

9.0 CALCULATIONS (DATA INTERPRETATION)

9.1 **Quantitative analysis**: When a compound is identified the quantitation of that compound is based on the integrated abundance of the primary ion. Quantitation is done with an internal standard. The internal standard used is the one nearest the retention time of the compound of interest.

9.1.1 **Water quantitation**:

$$\text{Concentration (ug/L)} = \frac{(Ax)(Is)(Vt)(Dil)}{(Ais)(RF_{avg})(Vo)Vi}$$

Where:

Ax	=	Area of primary ion for compound being measured
Is	=	Amount of internal standard injected (ng)
Vt	=	Volume of total extract (µL)
Ais	=	Area of primary ion of internal standard
RF_{avg}	=	Response factor of compound being measured
Vo	=	Volume of water extracted (ml)
Vi	=	Volume of extract injected (µL)
Dil	=	Dilution factor

9.1.2 **Sediment/ Soil Sludge (on dry weight basis) and waste (on wet weight basis):**

$$\text{Concentration (µg/Kg)} = \frac{(Ax)(Is)(Vt)(Dil)}{(Ais)(RF_{avg})(Vi)(Ws)(D)}$$

Where:

Ax, Is, Vt, Dil, Ais, RF_{avg}, and Vi are the same as in 9.1.1
Ws = Weight of sample extracted (grams)
D = % dry weight of sample. (1 for wet weight basis)

9.1.3 **Quantitation of Tentatively Identified Compounds**: Quantitation of a TIC is done assuming a response factor of one. The formulas above are used with the following modifications:

Ax = Area of the total ion chromatogram for compound being measured
Ais = Area of the total ion chromatogram for the internal standard

10.0 QUALITY ASSURANCE/QUALITY CONTROL

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10.1 The laboratory operates a quality control program to demonstrate the laboratory's capability and, with an ongoing analysis of spiked samples, to document the quality of the data generated. The ongoing data quality checks are compared with established

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performance criteria to determine if the result of the analysis meets the performance criteria of the method.

10.2 **Accuracy and Precision** All laboratory personnel must demonstrate initial proficiency for each sample preparation method/matrix that he or she performs. All new employees must successfully demonstrate initial proficiency prior to independently performing analysis on real samples. This must be accomplished by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The initial proficiency results will become part of each employee's training file.

QC Sample Preparation:
Spiking Solution: Four QC samples must be prepared from a spiking solution with the analytes of interest. The spiking solution should be made using standards **prepared independently from those used for calibration**. The samples must be prepared at a concentration that would result in data falling within the middle of the calibration curve, 50 µg/L. In most cases the blank spike or matrix spike solution is used. Prep: The samples are prepared in a clean matrix. In most cases this initial demonstration will simply be a matter of preparing four blank spikes with a batch of samples.

Sample Analysis:
The four QC samples must be analyzed within the criteria of the method being evaluated. The QC samples must be handled in exactly the same manner as actual samples.

Accuracy Calculation:
Accuracy is defined as the closeness of agreement between an observed value and an accepted reference value. Each of the four spiked samples will be calculated for percent recovery. The average of the percent recovery values is the accuracy result.

Precision Calculation:
Precision is defined as the agreement of a set of replicate measurements without assumption of knowledge of the true value. Precision is estimated by the relative standard deviation (RSD) of the four QC samples.

$$\%RSD = (s / \bar{x})\ 100\ \%$$

Where:

s = Standard Deviation of a finite number of values. On a scientific calculator use the **σxn-1** key.

$\bar{x}$ = The average of the four QC sample % recoveries.

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Reporting Accuracy and Precision Accuracy and Precision data should be presented with the following minimum info:

Matrix: Prep Method:
Clean-up Method: Analysis Method:

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Date Extracted:
Sample Prepared by: If Applicable
Sample Analyzed by:
Precision:

Date Analyzed:
Sample Fractionated by: If Applicable
Accuracy:

Parameter	*% Rec. QC 1*	*% Rec. QC 2*	*% Rec. QC 3*	*% Rec. QC 4*	*Average Recovery*	*Standard Deviation*	*%RSD*

Interpretation of Results:
The acceptance range for Accuracy is 45-135% with <30% RSD, 4 sporadic marginal exceedances are allowed. If any of the accuracy and precision results do not fall within the criteria then re-prep and reanalyze all QC samples only for those analytes outside criteria.

10.3 Method blanks are run with each batch to demonstrate that interferences from the analytical system, glassware, and reagents are under control. The blanks are carried through all stages of sample preparation. All target analytes should be less than ½ the MRL for the blank. See section 11.0 for corrective action for out of criteria results.

10.4 Perform DFTPP tune every 12 hours. Tuning acceptance criteria are presented in Table 2. The computer software will evaluate the tune information. The analyst should be aware of the process used. The tune *must* meet these criteria before sample analysis begins.

10.5 Run an initial calibration curve, using the primary source standards, each time major instrument maintenance occurs or if the CCV does not meet acceptance criteria. Acceptance criteria are presented in Section 8.6.

10.6 Run initial calibration verification (ICV) standard using secondary standards (7.2.5.3) following the initial calibration curve. Acceptance criteria are listed in Section 8.6.6. If the ICV does not meet criteria, re-analyze. If the second consecutive ICV does not meet criteria, then perform system maintenance and re-calibrate the system. The laboratory is allowed up to 5 sporadic marginal exceedances with the expanded criteria of 50-150%. ***NOTE: When analyzing samples for DoD, no sporadic marginal exceedances are allowed***

10.7 Run mid-point Continuing Calibration Verification (CCV) at 50 µg/L using the primary source standards on a daily basis before sample analysis. Also run a CCV every 12 hours during an analytical sequence. Acceptance criteria are listed in Section 8.7. See section 11.0 for corrective action for out of criteria results.

10.8 A blank spike and blank spike duplicate must be prepared and analyzed with each batch of samples. BS/BSDs are prepared using the second source standard and contain all target analytes. Control limits are 40-140% for the base/neutral compounds and 30-130% for the acid compounds. See Table 4 for DoD control limits. The BS/BSD should have a %RPD of ≤ 20% for waters and ≤ 30% for soils. If > 20% of all compounds fail or if >15% of any one category fails then re-extraction of the batch is necessary.

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Otherwise, note exceptions in the project narrative. ***DOD does not allow Sporadic Marginal Exceedances. See Table 4 for DoD blank spike acceptance criteria.***

10.8.1 Control charts will be maintained for the BS/BSD for a representative subset of target analytes and surrogate spikes. Annually, control limits will be determined for all target analytes and surrogates for comparison to default limits.

10.9 On an ongoing basis the laboratory analyzes matrix spikes and matrix spike duplicates from each batch of 20 samples. Matrix spike control limits are derived from BS/BSD samples, see section 10.8. For samples outside control limits, see Section 11.0 for corrective action.

10.10 Surrogates are added to all samples and QC samples. If the surrogate recoveries for customer or QC samples are outside control limits, see Section 11.0 for corrective action. Control limits for DoD surrogate are listed in Table 4.

Matrix	B/N Analytes	Acid Analytes
Soil	30-130%	30-130%
Water	30-130%	15-110%

10.11 The relative retention times (RRTs) need to be checked for each identified compound in samples, and compared to standard RRT. Acceptance criteria are presented in Section 11.0.

10.12 Internal standard area counts for standards and samples must meet specifications as described in Section 11.10.

10.13 Data shall be checked to ascertain if it conforms to accepted practices. All sample analytical results used for final data reporting must be between the low standard and the high standard. Results, which fall below the low standard or above the high standard, are to be reported as estimated values. Corrective actions are described in Section 11.0.

10.14 MDLs are determined in reagent water or organic-free sand/sodium sulfate and verified by thereafter by a quarterly MDL check (aka LOD Verification). See SOP 110_0013 for MDL/LOD/LOQ requirements. (Project-specific requirements may require that the MDL study be performed in the site-specific matrix.).

10.15 All manual integrations must be saved to PDF, when made, for verification. Refer to manual integration policy in SOP 110_0016.

11.0 DATA VALIDATION

11.1 Ensure that the DFTPP tune was run at the beginning of each 12-hour sequence for each batch of samples analyzed. The acceptance criteria are listed in Table 2.

11.1.1 If the DFTPP acceptance criteria are not met, perform any or all of the following corrective actions:

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Re-inject DFTPP.
Retune with PFTBA, and then re-inject DFTPP.
Clean MS source, retune with PFTBA, and re-inject DFTPP.

11.1.2 If the tuning criteria still cannot be met after performing the above, have the mass spectrometer serviced by manufacturer representative.

11.2 After an initial calibration curve is analyzed, ensure that the following criteria were met.

11.2.1 If the %RSD of any method target analyte is 20% or less, then the relative response factor is assumed to be constant over the calibration range, and the average relative response factor may be used for quantitation.

11.2.2 If the %RSD of any method target analyte is greater than 20%, calibration curves must be constructed using first or higher order regression fits. A minimum of 5 points must be used for linear regression and six points must be used for Quadratic Regression. The corresponding coefficient of determination (R^2) must be 0.99 or greater.

11.2.3 Minimum response factor criteria must be met per Table 6.

11.2.4 For the remaining compounds if greater than 20% of the compounds (15 for full list) have %RSD > 30, then recalibration is required.

11.2.5 If the acceptance criteria are not met, then the following corrective actions should be performed: (1) adjust the instrument and/or perform instrument maintenance; or (2) narrow the calibration range using six standards at different concentrations. The low end of the calibration curves must be carefully watched.

11.3 After an initial calibration (minimum of 5 point for average RF or linear regression, minimum of 6 points for quadratic regression) is performed, analyze initial calibration verification (ICV) standard. The percent difference must be within ±30% (±20% for DOD) for all target analytes with allowance for ***sporadic marginal exceedances (no allowances for DOD)***. Sporadic marginal exceedances have the expanded criterion of 40-160% drift. If criteria are not met, reanalyze the ICV or prepare a new calibration curve as necessary.

11.4 After the continuing calibration verification (CCV) standard is analyzed, ensure it was run at the required frequency (every 12 hours or initially before daily analysis). In addition, the following acceptance criteria must be met.

11.4.1 Check response factors. If the minimum response factors are not met, the system must be evaluated, and corrective action must be taken before sample analysis begins.

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11.4.2 All target compounds are evaluated to check the validity of the initial calibration with a limit of ≤20% drift or difference (allow up to 20% of compounds <40%D or up to 15% in each category). ***NOTE: For DoD samples, all compounds must have ≤20% drift, no allowances. DoD allows the use of a grand mean where the average of all compounds must be within ±20%, with no individual analytes (except CCCs) >25%.***

11.4.2.1 If these criteria are exceeded, corrective action is necessary. If corrective action fails to produce consecutive (immediate) calibration verification within acceptance criteria, then the analyst may perform maintenance to try to troubleshoot the instrument. After maintenance, the analyst must analyze two consecutive CCV at two concentration (one at or below mid-range) to demonstrate that corrective action was successful. If not, then a new initial calibration must be generated. These criteria must be met before sample analysis begins and re-analysis of samples up to the last acceptable CCV standard must occur with the following exceptions:

- When the acceptance criteria is exceeded high, high bias, then all samples that are non-detects may be reported.
- When the acceptance criteria for the CCV are exceeded low, low bias, sample results that exceed a maximum regulatory limit may be reported. **NOTE: When analyzing samples for DoD, all samples associated with a CCV below lower control limit must be re-analyzed.**

11.4.3 When performing analysis for MA MCP or DoD, any analyte outside of criteria in the CCV must be noted in the project narrative

11.5 Assess the method blank. The analyst must confirm that the method blank was analyzed at the required frequency and all target analytes are below the MRL (½ the MRL for DoD). Analytical batches with Method blanks outside acceptance criteria will be re-prepped and re-analyzed (where sample volume permits) with the following exceptions:

11.5.1 Samples that are at least ten times higher than the method blank may be reported, except for common lab contaminants, which must be less than five times the MRL.

11.5.2 When the method blank is less than 5% of the regulatory limit associated with the analyte the method blank would be acceptable.

11.5.3 If the analyte is found in the method blank above the MRL (½ the MRL for DoD) but is not in any of the associated samples, no corrective action is needed.

11.5.4 Any results that are reported with method blank contamination must be B-flagged.

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11.6 Assess that the matrix spike/matrix spike duplicates were analyzed at the required frequency. Acceptance criteria are listed in 10.9. If criteria are not met, perform the following corrective actions as appropriate.

11.6.1 If both BS/BSD and MS/MSD recoveries are unacceptable, then the entire batch of field and QC samples must be reanalyzed.

11.6.2 If the MS/MSD is unacceptable, but the BS/BSD is acceptable, then a potential matrix effect has been identified. Reasonable attempts must be made to address matrix interference. The client must be notified in the case narrative of the matrix problem.

11.7 Assess the surrogate recoveries for all samples, blanks, BS/BSD, MS, and MSD. The following acceptance criteria apply to surrogate recoveries.

11.7.1 The surrogate recoveries for all QC samples must be within control limits. If the BS/BSD and/or method blank recoveries are outside limits, re-analysis must be performed for verification. If still outside limits, then corrective action is to re-extract the batch.

11.7.2 If sample surrogate recoveries are outside control limits (10.10), the sample should be re-analyzed. If the results are still outside the limits, then re-prep and re-analyze the sample(s). An exception to this criterion is surrogates diluted out of sample due to matrix problems. In addition, if an obvious interference is present, the laboratory does not have to re-extract the sample but must provide the chromatogram with the project. *ESS Laboratory's policy allows for one acid and one base surrogate to be outside control limits before re-prepping sample batches (no allowance for DOD samples). If any surrogate is<10%, then sample must be re-prepped.*

11.8 The relative retention times must be checked for all identified compounds in both standards and samples. The internal standard absolute retention times must also be checked for all analyses. Acceptance criteria are as follows:

11.8.1 The relative retention times of each compound in each calibration and sample run should agree within 0.06 relative retention time (RRT) units. Late-eluting compounds usually have much better agreement.

11.8.2 Internal standard retention time:

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11.8.2.1 If the retention time for any CCV internal standard changes by more than 30 seconds from the **mid-point standard of the initial calibration**, the chromatographic system must be inspected for malfunctions and corrections must be made, as required.

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11.8.3 If the retention time for any sample internal standard changes by more than 30 seconds from the daily calibration, the chromatographic system must be inspected for malfunctions and corrections must be made, as required.

11.9 The analyst must verify that ion abundance meets specific criterion for the various analyses. The following acceptance criteria shall be checked for all appropriate samples.

11.9.1 All ions present in the standard mass spectrum at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) must be present in the sample spectrum.

11.9.2 The relative intensities of ions must agree within plus or minus 30% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample abundance must be between 20 and 80 percent.)

11.9.2.1 Molecular ions present in the reference spectrum should be present in the sample spectrum.

11.9.2.2 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.

11.9.2.3 Ions present in the reference spectrum, but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

11.9.3 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. When serving the role as QA (or referee) laboratory, tentatively identified compounds (TICs) are always reported. Computer-generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. For example, the RCRA permit or waste de-listing requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches will the mass spectral interpretation specialist assign a tentative identification. Guidelines for making tentative identification are as follows:

11.9.3.1 Relative intensities of major ions in the reference spectrum (ions > 10% of the most abundant ion) should be present in the sample spectrum.

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11.9.3.2 The relative intensities of the major ions should agree within ±20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70 %.)

11.9.3.3 Molecular ions present in the reference spectrum should be present in the sample spectrum. Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.

11.9.3.4 Ions present in the reference spectrum, but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

11.10 The analyst will check the internal standard area counts for all calibration standards, QC samples, and samples for quantitation. If the area for any of the CCV internal standards changes by a factor of two (-50% to +100%) from the mid-point standard of the initial calibration, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. All sample internal standards must be -50 to 200% of their associated CCV. If the internal standard area counts fail this criterion, the following corrective actions should be considered:

11.10.1 Check to ensure there was no error in internal standards preparation or addition. Also, check instrument performance.

11.10.2 If the CCV failed criterion, re-analyze once. If this second consecutive CCV still does not meet criterion, then a new initial calibration must be performed.

11.10.3 If the internal standards for samples are outside criterion, then re-analyze once. If re-analysis produces results within criteria, then report these results. If the re-analysis is still outside criterion, discuss matrix issue in project narrative. ***NOTE: If an obvious matrix effect is displayed on the chromatogram (Unresolved complex mixture, UCM), then re-analysis is not necessary. MA-MCP and CT RCP projects must have a copy of the chromatogram included with the report.***

11.11 The analyst must verify all reported results are derived from analytical results that are below the highest standard of the initial calibration curve and above the low standard. Values reported below the low standard are to be reported as estimated values (J values). For samples that exceed the calibration curve, dilute and analyze an appropriate sample aliquot.

11.12 All pertinent QA/QC information is to be reported in Attachment D. A second level of review must be performed by a second analyst and noted in Attachment D.

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12.0 REFERENCES

12.1 Methods SW846-8270C and D, Test Methods for the Analysis of Solid Waste.
12.2 HP GC EnviroQuant User's Guide, HPG1045A
12.3 HP Environmental Data Analysis User's Guide HPG0032C
12.4 HP 6890 GC Operations Manual
12.5 HP 5890 Series II Operations Manual
12.6 HP 5971A MS Operations Manual
12.7 HP 5973 MS Operations Manual
12.8 NELAC Quality Systems, Chapter 5, June 2003
12.9 DoD Quality Systems Manual, Final Version 4.1, April 2009
12.10 Massachusetts MCP WSC-CAM Section II B, Revision 1, Final.

13.0 POLLUTION PREVENTION and WASTE MANAGEMENT

13.1 ESS Laboratory's policies on pollution prevention and waste management are covered in SOP 90_0002, Hazardous Waste Contingency and Emergency Response Plan. All employees are trained in the requirements of the SOP.

14.0 METHOD PERFORMANCE

14.1 Precision and Accuracy data must be generated by all employees before performing this analysis on client samples. The data is generated by analyzing a method blank and four blank spike samples. Acceptance criteria are 45-135% Recovery and %RSD of ≤ 30%. Four (4) sporadic marginal exceedances are allowed.

14.2 The precision and accuracy data in Table 1 were obtained using the SOP. Values are in ug/L.

15.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

Table 1. Typical Precision and Accuracy data generated 1/3/2005

Compound Name	Spk Amt	Avg	RSD	%Rec	Compound Name	Spk Amt	Avg	RSD	%Rec
N-Nitrosodimethylamine	100	62.13	9	62.1	Acenaphthylene	100	64.46	5	64.5
Pyridine	100	33.39	55	33.4	2,6-Dinitrotoluene	100	85.33	11	85.3
2-Fluorophenol (Surr)	150	83.27	7	55.5	2-Nitroaniline	100	79.96	9	80.0
bis(2-Chloroethyl)ether	100	66.28	7	66.3	Acenaphthene	100	77.24	6	77.2
Phenol-d5 (Surr)	150	85.70	6	57.1	2,4-Dinitrophenol	100	49.15	20	49.1
2-Chlorophenol	100	66.38	6	66.4	Dibenzofuran	100	78.48	7	78.5
Phenol	100	61.65	5	61.6	4-Nitrophenol	100	80.25	10	80.2
Aniline	100	77.02	31	77.0	3-Nitroaniline	100	54.80	55	54.8
2-Chlorophenol-d4(Surr)	150	92.25	7	61.5	2,4-Dinitrotoluene	100	85.21	11	85.2
1,3-Dichlorobenzene	100	65.12	6	65.1	Fluorene	100	81.66	6	81.7
1,4-Dichlorobenzene	100	66.55	4	66.5	2,3,4,6-Tetrachlorophenol	100	88.20	8	88.2

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1,2 Dichlorobenzene-d4(Surr)	100	69.15	7	69.2	Diethylphthalate	100	82.05	7	82.1
1,2-Dichlorobenzene	100	69.31	4	69.3	4-Chloro-phenyl-phenyl ether	100	79.66	6	79.7
Benzyl Alcohol	100	78.72	7	78.7	4-Nitroaniline	100	68.82	21	68.8
bis(2-chloroisopropyl)ether	100	64.68	6	64.7	4,6-Dinitro-2-methylphenol	100	74.79	17	74.8
2-Methylphenol	100	66.15	7	66.1	n-Nitrosodiphenylamine	100	58.19	12	58.2
Acetophenone	100	75.74	6	75.7	Azobenzene	100	74.11	7	74.1
n-Nitroso-di-n-propylamine	100	74.40	7	74.4	2,4,6-Tribromophenol (Surr)	100	120.65	6	120.7
Hexachloroethane	100	68.22	6	68.2	4-Bromophenyl-phenylether	100	81.95	7	81.9
3+4-Methylphenol	100	69.03	7	69.0	Hexachlorobenzene	150	82.06	7	54.7
Nitrobenzene-d5 (Surr)	100	69.26	12	69.3	Pentachlorophenol	100	84.10	9	84.1
Nitrobenzene	100	67.77	11	67.8	Phenanthrene	100	77.83	7	77.8
Isophorone	100	68.67	11	68.7	Anthracene	100	76.01	7	76.0
2-Nitrophenol	100	71.52	10	71.5	Carbazole	100	71.52	8	71.5
Benzoic Acid	100	27.79	76	27.8	Di-n-butylphthalate	100	78.48	8	78.5
2,4-Dimethylphenol	100	56.93	26	56.9	Fluoranthene	100	82.08	8	82.1
bis(2-Chloroethoxy)methane	100	63.50	7	63.5	Benzidine	100	14.07	NA	NA
2,4-Dichlorophenol	100	72.11	11	72.1	Pyrene	100	75.55	7	75.6
1,2,4-Trichlorobenzene	100	68.79	10	68.8	Terphenyl-d14 (Surr)	100	80.18	7	80.2
Naphthalene	100	66.01	10	66.0	Butylbenzylphthalate	100	77.04	8	77.0
4-Chloroaniline	100	49.45	28	49.4	3,3'-Dichlorobenzidine	100	40.93	67	40.9
Hexachlorobutadiene	100	71.25	9	71.2	Benzo(a)anthracene	100	76.79	7	76.8
4-Chloro-3-methylphenol	100	73.73	11	73.7	Chrysene	100	77.87	8	77.9
2-Methylnaphthalene	100	67.72	11	67.7	bis(2-Ethylhexyl)phthalate	100	77.70	7	77.7
Hexachlorocyclopentadiene	100	8.96	16	9.0	Di-n-octylphthalate	100	86.23	7	86.2
2,4,6-Trichlorophenol	100	79.95	7	79.9	Benzo(b)fluoranthene	100	69.76	14	69.8
2,4,5-Trichlorophenol	100	82.78	6	82.8	Benzo(k)fluoranthene	100	121.30	21	121.3
2-Fluorobiphenyl (Surr)	100	78.33	8	78.3	Benzo(a)pyrene	100	75.50	8	75.5
Biphenyl	100	77.41	7	77.4	Indeno(1,2,3-cd)pyrene	100	88.02	14	88.0
2-Chloronaphthalene	100	78.08	6	78.1	Dibenzo(a,h)anthracene	100	83.84	12	83.8
Dimethylphthalate	100	79.39	8	79.4	Benzo(g,h,i)perylene	100	93.13	17	93.1

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TABLE 2
DFTPP KEY IONS AND ION CRITERIA

MASS	ION ABUNDANCE CRITERIA	MASS	ION ABUNDANCE CRITERIA
51	30% to 60% of mass 198	199	5% to 9% of mass 198
68	< 2% of mass 69	275	10% to 30% of mass 198
69	< 100% of mass 198	365	> 1% of mass 198
70	< 2% of mass 69	441	Present but less than mass 443
127	40% to 60% of mass 198		> 40% of mass 198
197	< 1% of mass 198	442	17% to 23% of mass 442
198	Base peak, 100% relative abundance	443	

Degradation of DDT to DDE and DDD should not exceed 20%. Benzidine and Pentachlorophenol should be present at their normal response and no peak tailing should be visible.

Table 3B
System Performance Check Compounds (SPCC)

N- Nitroso-di-n-propylamine
Hexachlorocyclopentadiene
2,4-Dinitrophenol
4-Nitrophenol

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Table 4

DoD Quality Systems Manual Blank Spike QC Limits

	DoD			
AQUEOUS				
Analyte	LCL	UCL	LMEL	UMEL
1,2,4-Trichlorobenzene	35	105	25	120
1,2-Dichlorobenzene	35	100	20	115
1,2-Diphenylhdrazine	55	115	45	120
1,3-Dichlorobenzene	30	100	20	110
1,4-Dichlorobenzene	30	100	20	110
2,4,5-Trichlorophenol	50	110	40	120
2,4,6-Trichlorophenol	50	115	40	125
2,4-Dichlorophenol	50	105	40	115
2,4-Dimethylphenol	30	110	15	125
2,4-Dinitrophenol	15	140	10	160
2,4-Dinitrotoluene	50	120	40	130
2,6-Dinitrotoluene	50	115	35	130
2-Chloronaphthalene	50	105	40	115
2-Chlorophenol	35	105	25	115
2-Methylnaphthalene	45	105	35	115
2-Methylphenol	40	110	25	120
2-Nitroaniline	50	115	35	125
2-Nitrophenol	40	115	25	125
3,3'-Dichlorobenzidine	20	110	10	125
3+4-Methylphenol	30	110	20	125
3-Nitroaniline	20	125	10	145
4,6-Dinitro-2-methylphenol	40	130	25	145
4-Bromophenyl-phenylether	50	115	40	125
4-Chloro-3-methylphenol	45	110	35	120
4-Chloroaniline	15	110	10	125
4-Chloro-phenyl-phenyl ether	50	110	40	120
4-Nitroaniline	35	120	20	130
4-Nitrophenol	0	125	0	145
Acenaphthene	45	110	35	120
Acenaphthylene	50	105	40	115
Anthracene	55	110	45	120
Benzo(a)anthracene	55	110	45	120
Benzo(a)pyrene	55	110	45	120
Benzo(b)fluoranthene	45	120	35	130
Benzo(g,h,i)perylene	40	125	25	135
Benzo(k)fluoranthene	45	125	30	135

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Benzoic Acid	0	125	0	150
Benzyl Alcohol	30	110	15	125
bis(2-Chloroethoxy)methane	45	105	35	115
bis(2-Chloroethyl)ether	35	110	25	120
bis(2-chloroisopropyl)ether	25	130	10	150
bis(2-Ethylhexyl)phthalate	40	125	30	140
Butylbenzylphthalate	45	115	35	130
Carbazole	50	115	35	130
Chrysene	55	110	45	120
Dibenzo(a,h)anthracene	40	125	30	140
Dibenzofuran	55	105	45	115
Diethylphthalate	40	120	30	130
Dimethylphthalate	25	125	10	145
Di-n-butylphthalate	55	115	45	125
Di-n-octylphthalate	35	135	20	155
Fluoranthene	55	115	45	125
Fluorene	50	110	40	120
Hexachlorobenzene	50	110	40	120
Hexachlorobutadiene	25	105	15	115
Hexachloroethane	30	95	15	105
Indeno(1,2,3-cd)pyrene	45	125	30	140
Isophorone	50	110	40	125
Naphthalene	40	100	30	115
Nitrobenzene	45	110	35	120
N-Nitrosodimethylamine	25	110	10	125
n-Nitroso-di-n-propylamine	35	130	20	145
n-Nitrosodiphenylamine	50	110	35	120
Pentachlorophenol	40	115	25	130
Phenanthrene	50	115	40	130
Phenol	0	115	0	135
Pyrene	50	130	35	140
Surrogates:				
2-Fluorobiphenyl	50	110	-	-
Terphenyl-d14	50	135	-	-
2,4,6-Tribromophenol	40	125	-	-
2-Fluorophenol	20	110	-	-
Phenol-d5/d6	10	115	0	135
Nitrobenzene-d5	40	110	-	-
SOIL				
Analyte	LCL	UCL	LMEL	UMEL
1,2,4-Trichlorobenzene	45	110	30	120
1,2-Dichlorobenzene	45	95	35	105
1,2-Diphenylhdrazine				

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1,3-Dichlorobenzene	40	100	30	110
1,4-Dichlorobenzene	35	105	25	115
2,4,5-Trichlorophenol	50	110	40	120
2,4,6-Trichlorophenol	45	110	30	120
2,4-Dichlorophenol	45	110	35	120
2,4-Dimethylphenol	30	105	20	115
2,4-Dinitrophenol	15	130	10	150
2,4-Dinitrotoluene	50	115	35	130
2,6-Dinitrotoluene	50	110	35	125
2-Chloronaphthalene	45	105	35	115
2-Chlorophenol	45	105	35	115
2-Methylnaphthalene	45	105	35	115
2-Methylphenol	40	105	30	115
2-Nitroaniline	45	120	30	130
2-Nitrophenol	40	110	30	120
3,3'-Dichlorobenzidine	10	130	0	145
3+4-Methylphenol	40	105	30	120
3-Nitroaniline	25	110	15	125
4,6-Dinitro-2-methylphenol	30	135	10	155
4-Bromophenyl-phenylether	45	115	35	130
4-Chloro-3-methylphenol	45	115	35	125
4-Chloroaniline	10	95	0	110
4-Chloro-phenyl-phenyl ether	45	110	35	120
4-Nitroaniline	35	115	20	125
4-Nitrophenol	15	140	10	160
Acenaphthene	45	110	35	120
Acenaphthylene	45	105	35	115
Anthracene	55	105	45	115
Benzo(a)anthracene	50	110	40	120
Benzo(a)pyrene	50	110	40	120
Benzo(b)fluoranthene	45	115	35	125
Benzo(g,h,i)perylene	40	125	25	140
Benzo(k)fluoranthene	45	125	30	135
Benzoic Acid	0	110	0	130
Benzyl Alcohol	20	125	10	140
bis(2-Chloroethoxy)methane	45	110	30	120
bis(2-Chloroethyl)ether	40	105	25	115
bis(2-chloroisopropyl)ether	20	115	10	130
bis(2-Ethylhexyl)phthalate	45	125	35	140
Butylbenzylphthalate	50	125	35	135
Carbazole	45	115	30	130
Chrysene	55	110	45	120
Dibenzo(a,h)anthracene	40	125	25	140

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Dibenzofuran	50	105	40	110
Diethylphthalate	50	115	40	125
Dimethylphthalate	50	110	40	120
Di-n-butylphthalate	55	110	45	120
Di-n-octylphthalate	40	130	25	145
Fluoranthene	55	115	45	125
Fluorene	50	110	40	115
Hexachlorobenzene	45	120	35	130
Hexachlorobutadiene	40	115	25	130
Hexachloroethane	35	110	20	120
Indeno(1,2,3-cd)pyrene	40	120	25	135
Isophorone	45	110	30	125
Naphthalene	40	105	30	120
Nitrobenzene	40	115	30	125
N-Nitrosodimethylamine	20	115	10	130
n-Nitroso-di-n-propylamine	40	115	30	125
n-Nitrosodiphenylamine	50	115	40	125
Pentachlorophenol	25	120	10	135
Phenanthrene	50	110	40	120
Phenol	40	100	30	110
Pyrene	45	125	35	135
Surrogates:				
2-Fluorobiphenyl	45	105	-	-
Terphenyl-d14	30	125	-	-
2,4,6-Tribromophenol	35	125	-	-
2-Fluorophenol	35	105	-	-
Phenol-d5/d6	40	100	-	-
Nitrobenzene-d5	35	100	-	-

%RPD for DoD.
LCL = Lower Control Limit
UCL = Upper Control Limit
LMEL = Lower Marginal Exceedance Limit
UMEL = Upper Marginal Exceedance Limit

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Table 5
Characteristic Ions for Semi-volatile Compounds
Page 1 of 3

Compound	Primary Ion	Secondary Ion(s)
Phenol	94	65, 66
Bis(2-chloroethyl)ether	93	63, 95
2-Chlorophenol	128	64, 130
1,3-Dichlorobenzene	146	148, 111
1,4-Dichlorobenzene-d_4 (I.S.)	152	150, 115
1,4-Dichlorobenzene	146	148, 111
Benzyl alcohol	108	79, 77
1,2-Dichlorobenzene	146	148, 111
N-Nitrosomethylethylamine	88	42, 88, 43, 56
Bis(2-chloroisopropyl)ether	45	77, 121
n-Nitroso-di-n-propylamine	70	42, 101, 130
Hexachloroethane	117	201, 199
Nitrobenzene	77	123, 65
Isophorone	82	95, 138
N-Nitrosodiethylamine	102	102, 42, 57, 44, 56
2-Nitrophenol	139	109, 65
2,4-Dimethylphenol	122	107, 121
Bis(2-chloroethoxy)methane	93	95, 123
Benzoic acid	122	105, 77
2,4-Dichlorophenol	162	164, 98
1,2,4-Trichlorobenzene	180	182, 145
Naphthalene-d_8 (I.S.)	136	68
Naphthalene	128	129, 127
Hexachlorobutadiene	225	223, 227
4-Chloro-3-Methylphenol	107	144, 142
2-Methylnaphthalene	142	141
2-Methylphenol	107	107, 108, 77, 79, 90
Hexachlorocyclopentadiene	237	235, 272
N-Nitrosopyrrolidine	100	100, 1, 42, 68, 69
Acetophenone	105	71, 105, 51, 120
4-Methylphenol	107	107, 108, 77, 79, 90

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Table 5
Characteristic IONS for Semivolatile Compounds
Page 2 of 3

Compound	Primary Ion	Secondary Ion(s)
2,4,6-Trichlorophenol	196	198, 200
2-Chloronaphthalene	162	127, 164
1-Chloronaphthalene	162	127, 164
2-Nitroaniline	65	92, 138
Dimethyl phthalate	163	194, 164
Acenaphthalene	152	151, 153
2,6-Dinitrotoluene	165	63, 89
3-Nitroaniline	138	108, 92
Acenaphthene-d_{10} (I.S.)	164	162, 160
Acenaphthene	154	153, 152
2,4-Dinitrophenol	184	63, 154
2,6-Dinitrophenol	162	162, 164, 126, 98, 63
4-Chloroaniline	127	127, 129, 65, 92
Dibenzofuran	168	139
4-Nitrophenol	139	109, 65
Diethyl phthalate	149	177, 150
Fluorene	166	165, 167
N-Nitrosodibutylamine	84	84, 57, 41, 116, 158
4-Chlorophenyl phenyl ether	204	206, 141
4,6-Dinitro-2-methylphenol	198	51, 105
N-Nitrosodiphenylamine	169	168, 167
2,4,5-Trichlorophenol	196	196, 198, 97, 132, 99
Hexachlorobenzene	284	142, 249
Pentachlorophenol	266	264, 268
4-Nitroaniline	138	138, 65, 108, 92, 80, 39
Phenanthrene-d_{10} (I.S.)	188	94, 80
Phenanthrene	178	179, 176
Anthracene	178	176, 179
1,4-Dinitrobenzene	168	168, 75, 50, 76, 92, 122
Di-n-butyl phthalate	149	150, 104
Fluoranthene	202	101, 203

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Table 5
Characteristic IONS for Semivolatile Compounds
Page 3 of 3

Compound	Primary Ion	Secondary Ion(s)
Pyrene	202	200, 203
Butyl benzyl phthalate	149	91, 206
4-Nitrobiphenyl	199	199, 152, 141, 169, 151
Benz(a)anthracene	228	229, 226
Chrysene-d_{12} (I.S.)	240	120, 236
3,3'Dichlorobenzidine	252	254, 126
Chrysene	228	226, 229
Bis(2-ethylhexyl)phthalate	149	167, 279
3,3-Dimethylbenzene	212	212, 106, 196, 180
Di-n-octyl phthalate	149	167, 43
Benzo(b)fluoranthene	252	253, 125
Benzo(k)fluoranthene	252	253, 125
Benzo(a)pyrene	252	253, 125
Perylene-d_{12} (I.S.)	264	260, 265
Indeno(1,2,3-cd)pyrene	276	138, 227
Dibenz(a,h)anthracene	278	139, 279
Benzo(g,h,i)perylene	276	138, 277
Pyridine	79	52
Aniline	93	62, 65
Biphenyl	154	153, 76
Carbazole	162	166, 139

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TABLE 6 (8270D Table4)

RECOMMENDED MINIMUM RESPONSE FACTOR CRITERIA FOR INITIAL AND CONTINUING CALIBRATION VERIFICATION USING THE SUGGESTED IONS FROM TABLE 1

Semivolatile Compounds	Minimum Response Factor (RF)
Benzaldehyde	0.010
Phenol	0.800
Bis(2-chloroethyl)ether	0.700
2-Chlorophenol	0.800
2-Methylphenol	0.700
2,2'-Oxybis-(1-chloropropane)	0.010
Acetophenone	0.010
4-Methylphenol	0.600
N-Nitroso-di-n-propylamine	0.500
Hexachloroethane	0.300
Nitrobenzene	0.200
Isophorone	0.400
2-Nitrophenol	0.100
2,4-Dimethylphenol	0.200
Bis(2-chloroethoxy)methane	0.300
2,4-Dichlorophenol	0.200
Naphthalene	0.700
4-Chloroaniline	0.010
Hexachlorobutadiene	0.010
Caprolactam	0.010
4-Chloro-3-methylphenol	0.200
2-Methylnaphthalene	0.400
Hexachlorocyclopentadiene	0.050
2,4,6-Trichlorophenol	0.200
2,4,5-Trichlorophenol	0.200
1,1'-Biphenyl	0.010
2-Chloronaphthalene	0.800

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TABLE 6
(continued)

Semivolatile Compounds	Minimum Response Factor (RF)
2-Nitroaniline	0.010
Dimethyl phthalate	0.010
2,6-Dinitrotoluene	0.200
Acenaphthylene	0.900
3-Nitroaniline	0.010
Acenaphthene	0.900
2,4-Dinitrophenol	0.010
4-Nitrophenol	0.010
Dibenzofuran	0.800
2,4-Dinitrotoluene	0.200
Diethyl phthalate	0.010
1,2,4,5-Tetrachlorobenzene	0.010
4-Chlorophenyl-phenyl ether	0.400
Fluorene	0.900
4-Nitroaniline	0.010
4,6-Dinitro-2-methylphenol	0.010
4-Bromophenyl-phenyl ether	0.100
N-Nitrosodiphenylamine	0.010
Hexachlorobenzene	0.100
Atrazine	0.010
Pentachlorophenol	0.050
Phenanthrene	0.700
Anthracene	0.700
Carbazole	0.010
Di-n-butyl phthalate	0.010
Fluoranthene	0.600
Pyrene	0.600
Butyl benzyl phthalate	0.010
3,3'-Dichlorobenzidine	0.010
Benzo(a)anthracene	0.800

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TABLE 6
(continued)

Semivolatile Compounds	Minimum Response Factor (RF)
Chrysene	0.700
Bis-(2-ethylhexyl)phthalate	0.010
Di-n-octyl phthalate	0.010
Benzo(b)fluoranthene	0.700
Benzo(k)fluoranthene	0.700
Benzo(a)pyrene	0.700
Indeno(1,2,3-cd)pyrene	0.500
Dibenz(a,h)anthracene	0.400
Benzo(g,h,i)perylene	0.500
2,3,4,6-Tetrachlorophenol	0.010

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16.0 DEFINITIONS

16.1 **Accuracy**: The closeness of agreement between an observed value and an accepted reference value. When applied to a set of observed values, accuracy will be a combination of a random component and of a common systematic error (or bias) component.

16.2 **Batch**: A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 20, then each group of 20 samples or less will all be handled as a separate batch.

16.3 **Bias**: The deviation due to matrix effects of the measured value (x_s - x_u) from a known spiked amount, where x_s is the spiked sample and x_u is the un-spiked sample. Bias can be assessed by comparing a measured value to an accepted reference value in a sample of known concentration or by determining the recovery of a known amount of contaminant spiked into a sample (matrix spike).

16.4 **Control Sample**: A QC sample introduced into a process to monitor the performance of the system.

16.5 **Equipment Blank**: A sample of analyte-free media which has been used to rinse the sampling equipment. It is collected after completion of decontamination and prior to sampling. This blank is useful in documenting adequate decontamination of sampling equipment.

16.6 **Method Reporting Limit**: The lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The MRL is generally 5 to 10 times the MDL. ESS Laboratory sets the MRL to the lowest non-zero standard in the calibration curve or higher.

16.7 **Field Duplicates**: Independent samples which are collected as close as possible to the same point in space and time. They are two separate samples taken from the same source, stored in separate containers, and analyzed independently. These duplicates are useful in documenting the precision of the sampling process.

16.8 **Blank Spike (BS)**: A known matrix spiked with compound(s) representative of the target analytes. This is used to document laboratory performance.

16.9 **Matrix**: The component or substrate (e.g., surface water, drinking water) which contains the analyte of interest.

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16.10 **Matrix Duplicate**: An intra-laboratory split sample which is used to document the precision of a method in a given sample matrix.

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16.11 **Matrix Spike**: An aliquot of sample spiked with a known concentration of target analyte(s). The spiking occurs prior to sample preparation and analysis. A matrix spike is used to document the bias of a method in a given sample matrix.

16.12 **Matrix Spike Duplicates**: Intra-laboratory split samples spiked with identical concentrations of target analyte(s). The spiking occurs prior to sample preparation and analysis. They are used to document the precision and bias of a method in a given sample matrix.

16.13 **Method Blank**: An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank should be carried through the complete sample preparation and analytical procedure. The method blank is used to document contamination resulting from the analytical process.

16.14 **Method Detection Limit (MDL)**: The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix type containing the analyte. See SOP 110_0013 for further explanation.

16.15 **Organic-Free Reagent Water**: For volatiles, all references to water in the method refer to water in which an interferant is not observed at the method detection limit of the compounds of interest. A water purification system is used to generate organic-free deionized water.

16.16 **Records**: Include all logbooks, papers, machine readable materials, or other documentary materials, regardless of physical form or characteristics.

16.17 **Surrogate**: An organic compound which is similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which is not normally found in environmental samples.

16.18 **Trip Blank**: A sample of analyte-free media taken from the laboratory to the sampling site and returned to the laboratory unopened. A trip blank is used to document contamination attributable to shipping and field handling procedures. This type of blank is useful in documenting contamination of volatile organics samples.

17.0 PERSONNEL QUALIFICATIONS

17.1 Analysts who perform this analysis must have a working knowledge or quantitative and qualitative analysis, instrumental methods of analysis, chemical laboratory methods, and equipment.

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17.2 All analysts, before performing any analysis, participate in the ESS Laboratory training program (SOP80_0016). The training process consists of reading the Standard Operating Procedure, gaining instruction on the procedure from an experienced analyst, and performing the initial demonstration of capability.

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18.0 TROUBLESHOOTING

18.1 **Daily Maintenance**: The following maintenance is performed prior to calibration or sample analysis. The purpose of this maintenance is to clean the injection system.

- Cool injection port and oven to room temperature.
- Turn off gas flow by setting EPC to 0.0. Access EPC by pressing the yellow shift key on the GC control panel then hitting the Injector B Temp button.
- Disconnect the column and cap the end with old septa.
- Remove septum nut and weldment.
- Take out liner and clean with methylene chloride using a cotton swab.
- Clean gold seal and inside the injection port.
- Re-attach the gold seal, replace liner and o-ring.
- Clean weldment Inlet and septum holder.
- Clean septum nut.
- Screw on weldment and replace septa if needed. The septa nut should be finger tight.
- Clip off about 6" of column and re-connect.
- Set EPC to 7.0, bake out injection port at 275°C and set oven temperature to 320°C for 30 minutes.
- Clean Injection syringe and guide
- Record daily maintenance in the Semi-Volatile maintenance logbook, Attachment D.

18.2 If DFTPP criteria are not met, the analysis must be repeated. Sample analysis can not begin until DFTPP meets acceptance criteria. Repeated failures indicate that the MS acquisition parameters must be adjusted. These parameters should be adjusted in manual tune, saved and the tune repeated.

18.3 If manual or auto-tune does not produce DFTPP spectra within acceptance criteria, the MS source may need cleaning. Cleaning instructions are found in the MS detector manual.

18.4 See laboratory supervisor or operations manager for all other maintenance problems.

18.5 Record all maintenance in the instrument's maintenance logbook.

19.0 DATA MANAGEMENT AND RECORDS

19.1 **Data Management** - ESS Laboratory utilizes the Promium Element LIMS system as part of its Data Management system. Client sample information is entered into

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ELEMENT LIMS and analyses are assigned to each sample. The LIMS allows EPA hold times, minimum batch QC requirements, and QC criteria to be assigned to each analysis. Standards can be entered and assigned to QC samples through the LIMS. Once analysis has been performed, data is imported using DataTool avoiding manual errors. In conjunction with Crystal Reports, the ELEMENT system allows for a wide variety of reporting formats.

19.2 **Records** – The specific retention periods required in the NELAC Standards, EPA-CFR and state and local statutes are followed or exceeded. At a minimum, data records are retained for five years from last use (10 years for drinking water). If there is a question about whether a record should be retained or disposed because no specific requirement could be found, the record is retained until such time as a retention period is specified. Records are stored in specified-labeled locations and are easily retrievable. All raw data associated with testing is also retained including; computer printouts, chromatograms, review forms, and logbooks.

20.0 ATTACHMENTS

Appendix A Low Level PAH and Pentachlorophenol analysis.

Appendix B Certificates of Analysis.

Appendix C Enviroquant Method.

Appendix D MS 1 & MS2 Sequence Logs and Maintenance Record.

Appendix E DOD Method Quality Objectives.

Appendix F MADEP CAM Method Quality Objectives.

Appendix G CPSC Phthalate Analysis procedure.

Appendix H 1,4-Dioxane Analysis using Isotope Dilution .

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SemiVolatile Organic Compounds Method 8270

Low Level PAH and Pentachlorophenol Analysis

SOP 60_8270C Appendix A

Method Summary: Aqueous samples are extracted by EPA Method 3510C. Extracts are concentrated to a final volume of 0.25 mls. Soil samples are extracted by EPA Methods 3541 or 3546 and concentrated to a final volume of 0.5 ml. Samples are analyzed by GC/MS in selective ion monitoring mode to achieve low detection limits. (0.05 µg/L for aqueous samples and 16.7 µg/Kg for soil samples).

The following are the 8270 modifications. Refer to SOP 60_ 8270 and appropriate preparation method SOP for all other procedures.:

Standards:
Stock: A 20ppm intermediate stock is prepared from the 200ppm 8270 stock by diluting 1ml to 10 ml with methylene chloride in a 10ml volumetric flask. This applies to the primary and second source standards.

Working Standards:

Standard	Amount of Stock Added (ul)	Final Volume ml	Final Conc. On Column ug/ml
Level 0.2	10	1	0.2
Level 0.4	20	1	0.4
Level 0.8	40	1	0.8
Level 1.0	50	1	1.0
Level 2.0	100	1	2.0
Level 4.0	200	1	4.0
Level 8.0	400	1	8.0
Level 10	500	1	10
Level 20	1000	1	20
SS PAH 1.0	50	1	1.0
SS PCP 5.0	250	1	5.0
CCV PAH 1.0	50	1	1.0
CCV PCP 5.0	250	1	5.0

a. Internal Standard: The 4000ppm 8270 standard is diluted 10x to 400ppm. (100µl to 1ml in methylene chloride).

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b. Surrogate Standard: A surrogate spiking solution is prepared at 2.5 µg/ml. Prepared by diluting 2.5 ml of the 8270 surrogate to 100 ml with acetone in a volumetric flask.
   i. Aqueous samples and QC spike volume is 250µl.
   ii. Soil Samples and QC spike is 500µl.

c. PAH matrix spike solution: Prepared at 2.5 µg/ml. Prepared by diluting 2.5 ml of the 8270 Matrix Spike to 100 ml with acetone in a volumetric flask
   i. Aqueous samples andQC spike 250µl.
   ii. Soil samples and QC spike 500µl.

d. Pentachlorophenol Spike: (Aqueous samples only) Use the SVOA matrix spike at 100µg/ml. (The high concentration standard maintains the integrity of the PCP) For aqueous samples spike 25µl into the BS/BSD/MS/MSD. By concentrating the extract to 0.25 ml, this will produce an on column concentration of 10µg/ml.

II. Extraction:

a. Aqueous: Up to 1L of sample is shaken by separatory funnel (3510C) with methylene chloride using a base -> acid extraction. The appropriate surrogates and spikes are added. The extract is concentrated to a final volume of 0.25ml in an NEVAP and measured with a 500 µl graduated Hamilton syringe. The extract is transferred to a microinsert in a GC target vial for analysis. The vial is capped. 1.25 µl of the 4000 ppm internal standard is spiked into the 250µl extract. (10µl is added to each 1ml standard.) This will produce a 20ppm on column concentration of Internal Standard.

b. Soil: 15grams is extracted by EPA Method 3541 or 3546. The Surrogate Spike (0.5 ml LL PAH SURR) and Matrix Spike (0.4 ml LL PAH MS) are added. The extract is concentrated to 0.5ml and transferred to a GC target vial and capped. 2.5µl of the 4000 ppm internal standard is spiked into each extract.

III. Analysis:

a. Tune: Performed in full scan mode. Ion ratios evaluated and must meet 8270 criteria. Analyzed prior to analysis, every 12 hours.

b. ICAL: All analytes <20% or Corr >= 0.99. Quad only acceptable for PCP and must be > 0.99 corr.

c. SCV: 70-130% recovery. No failures.

d. CCV: 70-130% No failures. Analyzed prior to sample analysis with no failures.

e. All analytes including surrogates and internal standards analyzed in selective ion monitoring mode. The following is a list of analytes and ions scanned:

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PAH SIMS

Group	Analytes	Type	Ions	Abs RT	Start Scan Time	Stop Scan Time
1	Baseline		74	1.00	1.00	
2	1,4-DCB-d4	IS	152,115,150	3.38	2.88	
	1,2-DCB-d4	Surr		3.56		
3	Nitrobenzene-d5	Surr	82,128,54	3.94	3.75	4.32
4	Napthalene-d8	IS	136,68,128,129	4.70	4.32	5.17
	Napthalene	Trgt		4.72		
5	2-Methylnaphthalene	Trgt	172,171,142,141	5.61	5.17	6.61
	1-Methylnaphthalene	Trgt		5.77		
	2-Fluorobiphenyl	Surr		6.19		
6	Acenaphthylene	Trgt	152,153,154,164	7.02	6.61	7.81
	Acenaphthene-d10	IS		7.29		
	Acenaphthene	Trgt		7.35		
7	Fluorene	Trgt	165,166,167	8.26	7.81	8.61
8	2,4,6-Tribromophenol	Surr	330,332	8.95	8.61	9.23
	Pentachlorophenol	Trgt		9.50		
9	Phenanthrene-d10	IS	176,178,188	10.08		
	Phenanthrene	Trgt	266,264	10.12		
	Anthracene	Trgt		10.21	9.23	11.40
10	Fluoranthene	Trgt	202,100,101	12.58	11.40	13.25
	Pyrene	Trgt		13.04		
11	Terphenyl-d14	Surr	122,212,244	13.46	13.25	14.53
12	Benzo(a)anthracene	Trgt	226,228,229,240	15.59	14.53	16.72
	Chrysene-d12	IS		15.62		
	Chrysene	Trgt		15.68		
13	Benzo(b)	Trgt	252,253,125,264	17.75	16.72	19.37
	Benzo(k)	Trgt		17.80		
	Benzo(a)pyrene	Trgt		18.34		
	Perylene-d12	IS		18.44		
14	Indeno 123	Trgt	276,278,138,139	20.29	19.37	to END
	Dibenzo(ah)	Trgt		20.31		
	Benzo(ghi)	Trgt		20.75		

IV: Reporting: On column results in µg/ml. Final results in ppb are calculated using initial and final volumes. See SOP 60_8270.

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Appendix B

Certificate of Analysis

OE05077-80

Semi-Volatiles Mixture

Product Number: SVM-8270 **Page:** 1 of 3

Lot Number: CG-0219 **Lot Issue Date:** Jan-2010 **Expiration Date:** Feb-2011

This Certified Reference Material (RM) was manufactured and verified in accordance with ULTRA's ISO 9001 registered quality system, and the analyte concentrations were verified by our ISO 17025 accredited laboratory. The true value and uncertainty value at the 95% confidence level for each analyte, determined gravimetrically, is listed below.

Analyte	CAS#	Analyte Lot	True Value
acenaphthene	000083-32-9	29697-41	1002 ± 5 µg/mL
acenaphthylene	000208-96-8	ER030707-01	1000 ± 5 µg/mL
anthracene	000120-12-7	33383-91	1002 ± 5 µg/mL
benz[a]anthracene	000056-55-3	ER121707-01	1002 ± 5 µg/mL
benzo[b]fluoranthene	000205-99-2	ER022008-02	1002 ± 5 µg/mL
benzo[k]fluoranthene	000207-08-9	34750-27	1002 ± 5 µg/mL
benzo[ghi]perylene	000191-24-2	ER041205-01	1002 ± 5 µg/mL
benzo[a]pyrene	000050-32-8	ER050707-01	1002 ± 5 µg/mL
carbazole	000086-74-8	CEE-III-129	1002 ± 5 µg/mL
chrysene	000218-01-9	ER081006-02	1002 ± 5 µg/mL
dibenz[a,h]anthracene	000053-70-3	ER091206-01	1002 ± 5 µg/mL
fluoranthene	000206-44-0	LB50809	1000 ± 5 µg/mL
fluorene	000086-73-7	04003CK	1002 ± 5 µg/mL
indeno[1,2,3-cd]pyrene	000193-39-5	ER082107-02	1002 ± 5 µg/mL
naphthalene	000091-20-3	14205KB	1001 ± 5 µg/mL
phenanthrene	000085-01-8	07419ME	1002 ± 5 µg/mL
pyrene	000129-00-0	01216PE	1002 ± 5 µg/mL
azobenzene	000103-33-3	04606DF	1003 ± 5 µg/mL
4-chloroaniline	000106-47-8	01310MW	1001 ± 5 µg/mL
2-chloronaphthalene	000091-58-7	FIE01	1001 ± 5 µg/mL

ULTRA uses balances calibrated with weights traceable to NIST in compliance with ANSI/NCSL Z-540-1 and ISO 9001, and calibrated Class A glassware in the manufacturing of these standards.

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William J. Leary
Quality Assurance Manager

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Certificate of Analysis

Semi-Volatiles Mixture

Product Number: SVM-8270 **Page:** 2 of 3

Lot Number: CG-0219 **Lot Issue Date:** Jan-2010 **Expiration Date:** Feb-2011

Analyte	CAS#	Analyte Lot	True Value
4-chloro-3-methylphenol	000059-50-7	061657	1000 ± 5 µg/mL
dibenzofuran	000132-64-9	480477	1001 ± 5 µg/mL
1,4-dichlorobenzene	000106-46-7	22628EB	1003 ± 5 µg/mL
2,4-dichlorophenol	000120-83-2	05729EZ	1001 ± 5 µg/mL
2-methyl-4,6-dinitrophenol	000534-52-1	168-60A	1003 ± 5 µg/mL
2,4-dinitrophenol	000051-28-5	12310CU	1000 ± 5 µg/mL
2,4-dinitrotoluene	000121-14-2	18219TA	1000 ± 5 µg/mL
2,6-dinitrotoluene	000606-20-2	08328CR	1003 ± 5 µg/mL
hexachlorobenzene	000118-74-1	02927MC	1002 ± 5 µg/mL
hexachloroethane	000067-72-1	12604HB	1004 ± 5 µg/mL
pentachlorophenol	000087-86-5	06324ED	1002 ± 5 µg/mL
2-nitrophenol	000088-75-5	N960045	1003 ± 5 µg/mL
4-nitrophenol	000100-02-7	LO1OL	1003 ± 5 µg/mL
2-nitroaniline	000088-74-4	13201TU	1004 ± 5 µg/mL
3-nitroaniline	000099-09-2	03020DL	1002 ± 5 µg/mL
4-nitroaniline	000100-01-6	3926DK	1002 ± 5 µg/mL
2,4,5-trichlorophenol	000095-95-4	07521CO	1004 ± 5 µg/mL
2,4,6-trichlorophenol	000088-06-2	07509ME	1004 ± 5 µg/mL
bis(2-chloroethyl) ether	000111-44-4	BC-013187	1004 ± 5 µg/mL
bis(2-chloroethoxy)methane	000111-91-1	381-90A	1002 ± 5 µg/mL
bis(2-ethylhexyl) phthalate	000117-81-7	D-23230	1002 ± 5 µg/mL
4-bromophenyl phenyl ether	000101-55-3	05819CO	1002 ± 5 µg/mL
butyl benzyl phthalate	000085-68-7	05613ED	1003 ± 5 µg/mL
4-chlorophenyl phenyl ether	007005-72-3	28314CA	1004 ± 5 µg/mL

ULTRA uses balances calibrated with weights traceable to NIST in compliance with ANSI/NCSL Z-540-1 and ISO 9001, and calibrated Class A glassware in the manufacturing of these standards.

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Semi-Volatiles Mixture

Product Number: SVM-8270 **Page:** 3 of 3

Lot Number: CG-0219 **Lot Issue Date:** Jan-2010 **Expiration Date:** Feb-2011

Analyte	CAS#	Analyte Lot	True Value
2-chlorophenol	000095-57-8	09106BQ	1000 ± 5 µg/mL
di-n-butyl phthalate	000084-74-2	AC-011687	1001 ± 5 µg/mL
1,2-dichlorobenzene	000095-50-1	08946KY	1003 ± 5 µg/mL
1,3-dichlorobenzene	000541-73-1	05902LZ	1003 ± 5 µg/mL
diethyl phthalate	000084-66-2	09615KD	1001 ± 5 µg/mL
2,4-dimethylphenol	000105-67-9	14822JF	1001 ± 5 µg/mL
dimethyl phthalate	000131-11-3	D-44220	1000 ± 5 µg/mL
di-n-octyl phthalate	000117-84-0	0001418030	1000 ± 5 µg/mL
hexachlorobutadiene	000087-68-3	339923/1	1002 ± 5 µg/mL
hexachlorocyclopentadiene	000077-47-4	03907AU	1000 ± 5 µg/mL
isophorone	000078-59-1	10830MQ	1002 ± 5 µg/mL
2-methylnaphthalene	000091-57-6	15416DA	1002 ± 5 µg/mL
nitrobenzene	000098-95-3	DI-10604DI	1001 ± 5 µg/mL
N-nitrosodimethylamine	000062-75-9	12070JS	1002 ± 5 µg/mL
N-nitrosodi-n-propylamine	000621-64-7	FIM01	1004 ± 5 µg/mL
1,2,4-trichlorobenzene	000120-82-1	00334TQ	1002 ± 5 µg/mL
o-cresol	000095-48-7	09416AA	1001 ± 5 µg/mL
p-cresol	000106-44-5	09410PI	1003 ± 5 µg/mL
bis(2-chloroisopropyl) ether	000108-60-1	PR-16041	1003 ± 5 µg/mL
phenol	000108-95-2	1373574	1004 ± 5 µg/mL

Matrix: methylene chloride/benzene (3:1)

ULTRA uses balances calibrated with weights traceable to NIST in compliance with ANSI/NCSL Z-540-1 and ISO 9001, and calibrated Class A glassware in the manufacturing of these standards.

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Quality Assurance Manager

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FOR LABORATORY USE ONLY-READ MSDS PRIOR TO USE.

Catalog No. : 563626 Lot No.: A066327

Description : Custom Appendix IX Mix #2

Expiration Date[1]: September 2010 Storage: Freezer

Handling: Sonicate ampule 15 minutes prior to opening

Elution Order	Compound	CAS #	Percent Purity [2]	Concentration [3] (weight/volume)	% Uncertainty [4] (95% C.L.; K=2)
1	1,4-Dioxane	123-91-1	99%	1,000.000 ug/ml	+/-0.64 %
2	Ethyl methacrylate	97-63-2	99%	1,000.000 ug/ml	+/-0.64 %
3	Methyl methanesulfonate	66-27-3	99%	1,000.000 ug/ml	+/-0.64 %
4	Ethyl methanesulfonate	62-50-0	99%	1,000.000 ug/ml	+/-0.64 %
5	Benzaldehyde	100-52-7	99%	1,000.000 ug/ml	+/-0.64 %
6	Pentachloroethane	76-01-7	98%	1,000.090 ug/ml	+/-0.64 %
7	Acetophenone	98-86-2	99%	1,000.000 ug/ml	+/-0.64 %
8	2,6-Dichlorophenol	87-65-0	99%	1,000.000 ug/ml	+/-0.64 %
9	Hexachloropropene	1888-71-7	99%	1,000.000 ug/ml	+/-0.64 %
10	epsilon-Caprolactam	105-60-2	99%	1,000.000 ug/ml	+/-0.64 %
11	Isosafrole (cis & trans)	120-58-1	99%	1,000.000 ug/ml	+/-0.64 %
12	1,2,4,5-Tetrachlorobenzene	95-94-3	99%	1,000.000 ug/ml	+/-0.64 %
13	Safrole	94-59-7	99%	1,000.000 ug/ml	+/-0.64 %
14	Biphenyl	92-52-4	99%	1,000.000 ug/ml	+/-0.64 %
15	1-Chloronaphthalene	90-13-1	86%	1,000.180 ug/ml	+/-0.64 %
16	Diphenyl ether	101-84-8	99%	1,000.000 ug/ml	+/-0.64 %
17	1,4-Naphthoquinone	130-15-4	99%	1,000.000 ug/ml	+/-0.64 %
18	Pentachlorobenzene	608-93-5	99%	1,000.000 ug/ml	+/-0.64 %
19	Diallate (cis and trans)	2303-16-4	99%	1,000.000 ug/ml	+/-0.64 %
20	Phenacetin	62-44-2	99%	1,000.000 ug/ml	+/-0.64 %
21	Atrazine	1912-24-9	99%	1,000.000 ug/ml	+/-0.64 %
22	Pentachloronitrobenzene (quintozene)	82-68-8	99%	1,000.000 ug/ml	+/-0.64 %
23	Pronamide (Propyzamide)	23950-58-5	98%	1,000.090 ug/ml	+/-0.64 %
24	4-Nitroquinoline-N-oxide	56-57-5	----%	1,000.000 ug/ml	+/-0.64 %
25	Isodrin	465-73-6	98%	1,000.090 ug/ml	+/-0.64 %
26	Aramite	140-57-8	----%	1,000.000 ug/ml	+/-0.64 %
27	Chlorobenzilate	510-15-6	99%	1,000.000 ug/ml	+/-0.64 %
28	Kepone	143-50-0	99%	1,000.000 ug/ml	+/-0.64 %
29	7,12-Dimethylbenz(a)anthracene	57-97-6	99%	1,000.000 ug/ml	+/-0.64 %
30	3-Methylcholanthrene	56-49-5	99%	1,000.000 ug/ml	+/-0.64 %
31	Dibenz(a,j)acridine	224-42-0	99%	1,000.000 ug/ml	+/-0.64 %

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FOR LABORATORY USE ONLY-READ MSDS PRIOR TO USE.

Catalog No. : 31850 Lot No.: A070659

Description : 8270 MegaMix

Expiration Date[1]: April 2011 Storage: Freezer

Handling: Sonicate ampule 15 minutes prior to opening

Elution Order	Compound	CAS #	Percent Purity [2]	Concentration [3] (weight/volume)	% Uncertainty [4] (95% C.L.; K=2)
1	N-Nitrosodimethylamine	62-75-9	99%	1,000.000 ug/ml	+/-0.60 %
2	Pyridine	110-86-1	99%	1,000.000 ug/ml	+/-0.60 %
3	Aniline	62-53-3	99%	1,000.000 ug/ml	+/-0.60 %
4	Phenol	108-95-2	99%	1,000.000 ug/ml	+/-0.60 %
5	Bis(2-chloroethyl)ether	111-44-4	99%	1,000.000 ug/ml	+/-0.60 %
6	2-Chlorophenol	95-57-8	99%	1,000.000 ug/ml	+/-0.60 %
7	1,3-Dichlorobenzene	541-73-1	99%	1,000.000 ug/ml	+/-0.60 %
8	1,4-Dichlorobenzene	106-46-7	99%	1,000.000 ug/ml	+/-0.60 %
9	Benzyl alcohol	100-51-6	99%	1,000.000 ug/ml	+/-0.60 %
10	1,2-Dichlorobenzene	95-50-1	99%	1,000.000 ug/ml	+/-0.60 %
11	2-Methylphenol (o-cresol)	95-48-7	99%	1,000.000 ug/ml	+/-0.60 %
12	Bis(2-chloroisopropyl)ether	108-60-1	99%	1,000.000 ug/ml	+/-0.60 %
13	4-Methylphenol (p-cresol)	106-44-5	99%	500.000 ug/ml	+/-0.60 %
14	3-Methylphenol (m-cresol)	108-39-4	99%	500.000 ug/ml	+/-0.60 %
15	N-Nitroso-di-n-propylamine	621-64-7	99%	1,000.000 ug/ml	+/-0.60 %
16	Hexachloroethane	67-72-1	99%	1,000.000 ug/ml	+/-0.60 %
17	Nitrobenzene	98-95-3	99%	1,000.000 ug/ml	+/-0.60 %
18	Isophorone	78-59-1	99%	1,000.000 ug/ml	+/-0.60 %
19	2-Nitrophenol	88-75-5	99%	1,000.000 ug/ml	+/-0.60 %
20	2,4-Dimethylphenol	105-67-9	99%	1,000.000 ug/ml	+/-0.60 %
21	Bis(2-chloroethoxy)methane	111-91-1	99%	1,000.000 ug/ml	+/-0.60 %
22	2,4-Dichlorophenol	120-83-2	99%	1,000.000 ug/ml	+/-0.60 %
23	1,2,4-Trichlorobenzene	120-82-1	97%	999.973 ug/ml	+/-0.60 %
24	4-Chloroaniline	106-47-8	99%	1,000.000 ug/ml	+/-0.60 %
25	Naphthalene	91-20-3	99%	1,000.000 ug/ml	+/-0.60 %
26	Hexachlorobutadiene	87-68-3	98%	1,000.029 ug/ml	+/-0.60 %
27	4-Chloro-3-methylphenol	59-50-7	99%	1,000.000 ug/ml	+/-0.60 %
28	2-Methylnaphthalene	91-57-6	97%	1,000.070 ug/ml	+/-0.60 %
29	1-Methylnaphthalene	90-12-0	99%	1,000.000 ug/ml	+/-0.60 %
30	Hexachlorocyclopentadiene	77-47-4	99%	1,000.000 ug/ml	+/-0.60 %
31	2,4,6-Trichlorophenol	88-06-2	99%	1,000.000 ug/ml	+/-0.60 %
32	2,4,5-Trichlorophenol	95-95-4	99%	1,000.000 ug/ml	+/-0.60 %
33	2-Chloronaphthalene	91-58-7	99%	1,000.000 ug/ml	+/-0.60 %
34	2-Nitroaniline	88-74-4	99%	1,000.000 ug/ml	+/-0.58 %
35	1,4-Dinitrobenzene	100-25-4	99%	1,000.000 ug/ml	+/-0.60 %
36	Acenaphthylene	208-96-8	99%	1,000.000 ug/ml	+/-0.60 %
37	1,3-Dinitrobenzene	99-65-0	99%	1,000.000 ug/ml	+/-0.60 %
38	Dimethylphthalate	131-11-3	99%	1,000.000 ug/ml	+/-0.60 %
39	2,6-Dinitrotoluene	606-20-2	99%	1,000.000 ug/ml	+/-0.60 %
40	1,2-Dinitrobenzene	528-29-0	99%	1,000.000 ug/ml	+/-0.60 %
41	Acenaphthene	83-32-9	99%	1,000.000 ug/ml	+/-0.60 %
42	3-Nitroaniline	99-09-2	99%	1,000.000 ug/ml	+/-0.58 %
43	2,4-Dinitrophenol	51-28-5	97%	1,000.009 ug/ml	+/-0.60 %
44	Dibenzofuran	132-64-9	99%	1,000.000 ug/ml	+/-0.60 %
45	2,4-Dinitrotoluene	121-14-2	99%	1,000.000 ug/ml	+/-0.60 %
46	4-Nitrophenol	100-02-7	99%	1,000.000 ug/ml	+/-0.60 %

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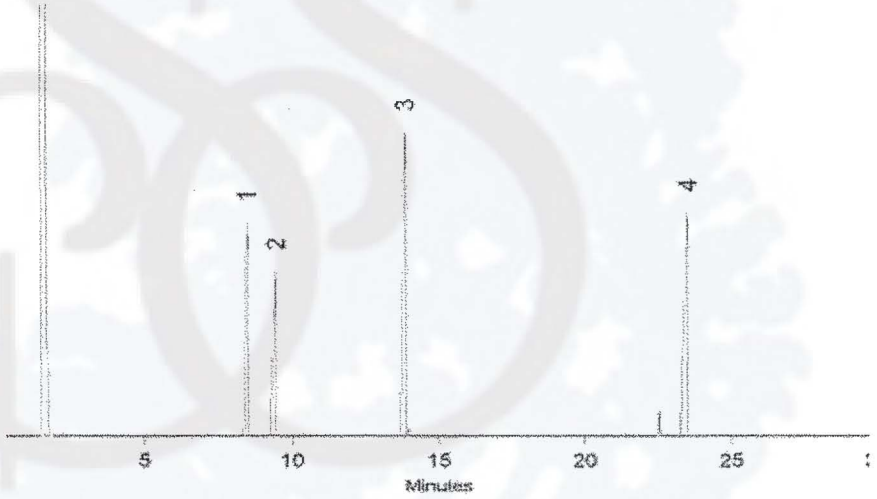
FOR LABORATORY USE ONLY-READ MSDS PRIOR TO USE.

Catalog No. : 31082 **Lot No.:** A062485
Description : B/N Surrogate Standard Mix (3/90)
Expiration Date[1]**:** September 2011 **Storage:** Refrigerate

Elution Order	Compound	CAS #	Percent Purity [2]	Concentration [3] (weight/volume)	Percent Uncertainty [4]
1	1,2-Dichlorobenzene-d4	2199-69-1	99%	5,000.000 ug/ml	+/-0.03 %
2	Nitrobenzene-d5	4165-60-0	99%	5,000.000 ug/ml	+/-0.03 %
3	2-Fluorobiphenyl	321-60-8	99%	5,000.000 ug/ml	+/-0.03 %
4	p-Terphenyl-d14	1718-51-0	97%	4,999.962 ug/ml	+/-0.03 %
Solvent:	Methylene Chloride	75-09-2	99%		

Column: 30m x .25mm x .5um
Rtx-5 (cat.#10238)
Carrier Gas: hydrogen @ 40cm/sec.
Temp. Program: 100°C to 330°C
@ 20°C/min. (hold 5 min.)
Inj. Temp: 250°C
Det. Temp: 300°C
Det. Type: FID



Diane Shaffer
Diane Shaffer - QA Analyst

Balance 1128353505

Manufactured under Restek's ISO 9001:2000
Registered Quality System
Certificate #FM80397

1 Expiration date of the unopened ampul stored at recommended temperature.
2 Purity was determined by one or more of the following techniques: GC/FID, HPLC, GC/ECD, GC/MS. Value rounded to the nearest LOWER whole percentage. In addtion to detectors listed above, chemical identity and purity are confirmed using 1 or more of the following: MS, DSC, solid probe MS, GC/FPD, GC/NPD, GC/TC, FTIR, melting point, refractive index, and Karl Fisher. See data pack or contact Restek for further details.
3 Based upon gravimetric preperation with balance calibration verified using NISTtraceable weights (seven mass levels).
4 Percent Uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.

Tech Tip:

Due to the limited solubility of p-terphenyl-d14 in methanol, we do not recommend that this mixture be diluted in methanol.

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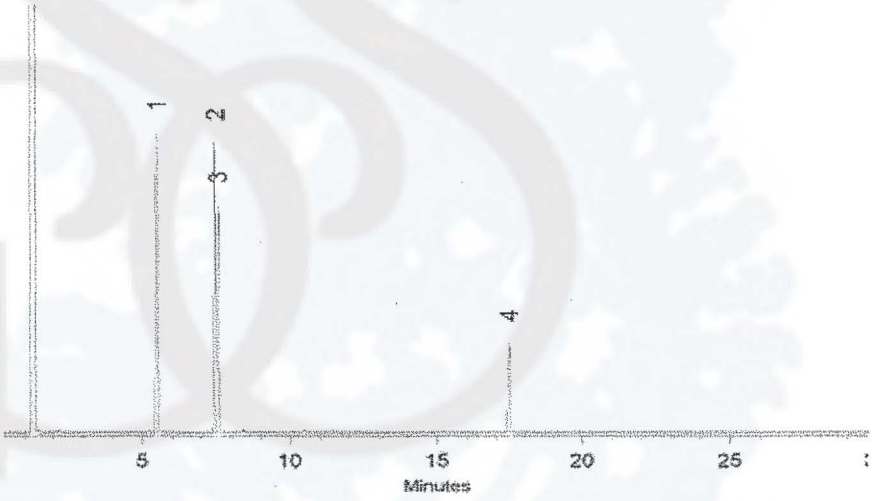
FOR LABORATORY USE ONLY-READ MSDS PRIOR TO USE.

Catalog No. : 31083 Lot No.: A061440
Description : Acid Surrogate Standard Mix (3/90)
Expiration Date[1]: January 2013 Storage: Refrigerate

Elution Order	Compound	CAS #	Percent Purity [2]	Concentration [3] (weight/volume)	Percent Uncertainty [4]
1	2-Fluorophenol	367-12-4	99%	7,500.100 ug/ml	+/-0.03 %
2	Phenol-d6	13127-88-3	99%	7,502.200 ug/ml	+/-0.03 %
3	2-Chlorophenol-d4	93951-73-6	99%	7,500.900 ug/ml	+/-0.03 %
4	2,4,6-Tribromophenol	118-79-6	99%	7,500.000 ug/ml	+/-0.03 %
Solvent:	Methanol	67-56-1	99%		

Column: 30m x .25mm x .25um
Rtx-5 (cat.#10223)
Carrier Gas: hydrogen @ 40cm/sec.
Temp. Program: 40°C (hold 2 min.) to 330°C
@ 10°C/min. (hold 10 min.)
Inj. Temp: 250°C
Det. Temp: 300°C
Det. Type: FID



Diane Shaffer
Diane Shaffer - QA Analyst

Balance 1128360905

Manufactured under Restek's ISO 9001:2000
Registered Quality System
Certificate #FM80397

1 Expiration date of the unopened ampul stored at recommended temperature.
2 Purity was determined by one or more of the following techniques: GC/FID, HPLC, GC/ECD, GC/MS. Value rounded to the nearest LOWER whole percentage. In addition to detectors listed above, chemical identity and purity are confirmed using 1 or more of the following: MS, DSC, solid probe MS, GC/FPD, GC/NPD, GC/TC, FTIR, melting point, refractive index, and Karl Fisher. See data pack or contact Restek for further details.
3 Based upon gravimetric preperation with balance calibration verified using NIST traceable weights (seven mass levels).
4 Percent Uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.

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Website AccuStandard.com

CERTIFICATE OF ANALYSIS

CATALOG NO: DRH-006S
DESCRIPTION: Proposed DEP(MA) - PAH Mix
LOT: 209101293
SOLVENT: CH2Cl2

EXPIRATION: Oct 29, 2019

See reverse for additional certification information.

Component	CAS #	Purity % (GC/MS)	Prepared Concentration[1] (µg/mL)	Certified Analyte Concentration[2] (µg/mL)
Acenaphthene	83-32-9	100	1005	1005
Acenaphthylene	208-96-8	99.1	1000	991
Anthracene	120-12-7	100	1001	1001
Benz(a)anthracene	56-55-3	99.6	1000	996
Benzo(a)pyrene	50-32-8	98.9	1002	991
Benzo(b)fluoranthene	205-99-2	100	1004	1004
Benzo(g,h,i)perylene	191-24-2	99.6	1002	998
Benzo(k)fluoranthene	207-08-9	99.5	1000	995
Chrysene	218-01-9	100	1005	1005
Dibenz(a,h)anthracene	53-70-3	99.5	1006	1001
Fluoranthene	206-44-0	99.9	1001	1000
Fluorene	86-73-7	98.1	1007	988
Indeno(1,2,3-cd)pyrene	193-39-5	98.2	1006	988
2-Methylnaphthalene	91-57-6	100	1004	1004
Naphthalene	91-20-3	99.8	1004	1002
Phenanthrene	85-01-8	98.9	1002	991
Pyrene	129-00-0	98.6	1006	992

17 Components

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1. All weights are traceable through NIST, Test No. 822/272103-05
2. Certified Analyte Concentration = Purity x Prepared Concentration. The Uncertainty calculated for this product is ±2% which is the Combined Uncertainty uc(y). It represents an estimated standard deviation equal to the positive square root of the total variance of the uncertainty of components. The Expanded Uncertainty is U which is Uc(y) *K where K is the coverage factor at the 95% confidence level (K=2).
3. A product with a suffix (-1A, -2B, etc. or -01, -02, etc.) on its lot# has had its expiration date extended and is identical to the same lot# without the suffix.

Certified by: R. Cooper

AccuStandard is accredited to ISO/IEC 17025:2005 and certified to ISO 9001:2000

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9K24069

DESCRIPTION: Pentachlorophenol

CATALOG NO.: 48692 MFG DATE: Sep-2008

LOT NO.: LB61534 EXPIRATION DATE: Sep-2011

SOLVENT: METHANOL

ANALYTE	CAS NUMBER	PERCENT PURITY(1)	WEIGHT(2) CONCENTRATION	ANALYTICAL(3)		STD DEV	SUPELCO LOT NO
PENTACHLOROPHENOL	87-86-5	98.9	500.5	507.0	+/-	11.92	LB37346

(1) Determined by capillary GC-FID, unless otherwise noted.

(2) NIST traceable weights are used to verify balance calibration with the preparation of each lot. Concentration of analyte in solution is ug/ml +/- 0.5%, uncertainty based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 98% pure.

(3) Determined by chromatographic analysis against an independently prepared reference lot. Mean of replicate injections.

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Elwood Doughty
Quality Control Supervisor

Supelco warrants that its products conform to the information contained in this publication. Purchaser must determine the suitability of the product for its particular use. Please see the latest catalog or order invoice and packing slip for additional terms and conditions of sale.

SUPELCO Analytical
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Bellefonte, PA 16823-0048 USA
Phone (814) 359-3441

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TOPLEVEL PARAMETERS

(GC/MS-1)

Method Information For: C:\HPCHEM\1\METHODS\SV1NB.M

Method Sections To Run:

Appendix C

( ) Save Copy of Method With Data
Pre-Run Cmd/Macro =
(X) Data Acquisition
( ) Data Analysis
( ) Post-Run Cmd/Macro =

Method Comments:
THIS METHOD IS USED TO ANALYZE FOR BASE-NEUTRAL AND ACID EXTRACTABLE COMPOUNDS USING A CAPILLARY COLUMN. THIS METHOD IS USING TCL SOW 88 LIST. 625 AND 8270 ARE APPLICABLE 02/95.

END OF TOPLEVEL PARAMETERS

ACQUISITION PARAMETERS

General Information

Inlet : GC
Tune File : DFTPP.U
Acquisition Mode : Scan

MS Information

Solvent Delay : 0.75 min

EM Absolute : False
EMV Offset : 0.0
Resulting Voltage : 1835.3

[Scan Parameters]

Low Mass : 35
High Mass : 500
Threshold : 500
Sampling # : 2 A/D Samples 4
Plot 1 low mass : 35
Plot 1 high mass: 500

[Real Time Plot Parameters]

Time Window : 25 min
Iconize Real Time Display : False
Plot 1 type : Extracted ion
Scale minimum : 0
Scale maximum : 400000
Plot 2 type : No plot

GC Inlet Information

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[Inlet A Temperature Program Information]

Oven Track : Off
Inlet A Off

[Inlet B Temperature Program Information]

Oven Track : Off
Initial Temp. : 275 C
Initial Time : 480.00 min

Level	Rate (C/min)	Final Temp. (C)	Final Time (min)
1	0		

Total Program Time: 480.00 min

[Inlet A Pressure Program Information]

Constant Flow : On 7.1 psi at 40 C
Pressure Units : psi

[Inlet A Flow Settings]

Column length : 30.00 m
Column diameter : 0.250 mm
Gas : He
Vacuum compensation : On
Pressure : 8.0 psi
Flow : 1.1 ml/min
Linear velocity : 37.5 cm/sec
Split flow : 1 ml/min
Split ratio : 0.9

[Inlet B Pressure Program Information]

Constant Flow : Off
Initial Pres. : 7.1 psi
Initial Time : 0.00 min

Level	Rate(psi/min)	Final Pres.(psi)	Final Time (min)
1	99.00	36.0	0.70
2	99.00	7.1	0.00
3	0		

Total Program Time: 1.28 min
Pressure Units : psi

[Inlet B Flow Settings]

Column length : 30.00 m
Column diameter : 0.250 mm
Gas : He
Vacuum compensation : On
Pressure : 7.1 psi
Flow : 0.9 ml/min
Linear velocity : 33.9 cm/sec
Split flow : 1 ml/min
Split ratio : 1.2

MASTER

[Auxiliary Channel C Information]

Comment:

Pressure Program:
Initial Pres. : 0.0 psi
Initial Time : 480.00 min

Level	Rate(psi/min)	Final Pres.(psi)	Final Time (min)

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```
  1                0
Total Program Time: 480.00 min
```

[Auxiliary Channel D Information]

```
Comment:

Pressure Program:
Initial Pres. : 0.0 psi
Initial Time  : 480.00 min

Level     Rate(psi/min)   Final Pres.(psi)  Final Time (min)
  1               0
Total Program Time: 480.00 min
```

[Auxiliary Channel E Information]

```
Comment:

Pressure Program:
Initial Pres. : 0.0 psi
Initial Time  : 480.00 min

Level     Rate(psi/min)   Final Pres.(psi)  Final Time (min)
  1               0
Total Program Time: 480.00 min
```

[Auxiliary Channel F Information]

```
Comment:

Pressure Program:
Initial Pres. : 0.0 psi
Initial Time  : 480.00 min

Level     Rate(psi/min)   Final Pres.(psi)  Final Time (min)
  1               0
Total Program Time: 480.00 min
```

GC Temperature Information

[GC Zone Temperatures]

```
Inj. A : 250 C  Off
Inj. B : 275 C
Det. A : 50 C  Off
Det. B : 300 C
Aux.   : 280 C  Off
```

[Oven Parameters]

```
Oven Equib Time  : 0.20 min
Oven Max         : 325 C
Oven             : On
Cryo             : Off
Ambient          : 25 C
Cryo Blast       : Off
```

[Oven Program]

```
Initial Temp. : 60 C
Initial Time  : 2.00 min
```


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Level	Rate (C/min)	Final Temp. (C)	Final Time (min)
1	35.00	130	0.00
2	12.00	325	3.75
3	0.00		

Next Run Time : 24.00 min

Injector Information
-------- -----------

Injection Source : Auto
Injection Location : Front

Sample Washes : 1
Sample Pumps : 4
Sample Volume : 1 stop(s)
Viscosity Delay : 1 sec
Solvent A Washes : 4
Solvent B Washes : 2
On Column : No

[Purge Information]

Purge A/B	Init. Value	On Time	Off Time
A	On	0.15	0.00
B	On	0.15	0.00

END OF ACQUISITION PARAMETERS

DATA ANALYSIS PARAMETERS

Method Name: C:\HPCHEM\1\METHODS\SV1NB.M

Percent Report Settings

Sort By: Signal

Output Destination
Screen: Yes
Printer: No
File: No

Integration Events: Meth Default

Generate Report During Run Method: No

Signal Correlation Window: 0.020

Qualitative Report Settings

Peak Location of Unknown: Apex

Library to Search	Minimum Quality
C:\DATABASE\NBS75K.L	0
C:\DATABASE\PAH.L	0

Integration Events: Meth Default

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Report Type: Summary

Output Destination
    Screen: No
    Printer: Yes
    File: No

Generate Report During Run Method: No

Quantitative Report Settings

Report Type: Detailed

Output Destination
    Screen: No
    Printer: Yes
    File: No

Generate Report During Run Method: No

ELEMENT ID: 0603035(SOIL) 0603036(AQUEOUS)
Calibration Last Updated: Fri Apr 07 09:13:18 2006

Reference Window: 5.00 Percent
Non-Reference Window: 5.00 Percent
Correlation Window: 0.03 minutes
Default Multiplier: 1.00
Default Sample Concentration: 0.00

Compound Information

---

1) 1,4-Dichlorobenzene-d4 (ISTD)

Ret. Time 3.86 min., Extract & Integrate from 3.36 to 4.36 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	152.00			*** METH DEFAULT ***
Q1	115.00	58.70	30.0	*** METH DEFAULT ***
Q2	150.00	289.20	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ng/uL)	Response
5		40.000	442167
10		40.000	372682
50		40.000	381573
80		40.000	339990
120		40.000	337070
160		40.000	306591
200		40.000	356509
25		40.000	437292
CC		40.000	499862

Qualifier Peak Analysis ON ISTD conc: 40.000 ng/uL
Curve Fit: Avg. RF

---

2) N-Nitrosodimethylamine ( )

Ret. Time 0.85 min., Extract & Integrate from 0.35 to 1.35 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	74.05			*** METH DEFAULT ***
Q1	42.10	53.30	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ng/uL)	Response

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TOPLEVEL PARAMETERS

Method Information For: C:\HPCHEM\1\METHODS\SV2KC.M

Method Sections To Run:

( ) Save Copy of Method With Data
( ) Pre-Run Cmd/Macro =
(X) Data Acquisition
( ) Data Analysis
( ) Post-Run Cmd/Macro =

Appendix C

Method Comments:
8270

END OF TOPLEVEL PARAMETERS

INSTRUMENT CONTROL PARAMETERS (MS2)

Sample Inlet: GC
Injection Source: GC ALS
Mass Spectrometer: Enabled

7673 Injector

Front Injector:
No parameters specified

Back Injector:

Sample Washes	2
Sample Pumps	2
Injection Volume	1.0 microliters
Syringe Size	10.0 microliters
On Column	Off
Nanoliter Adapter	Off
PostInj Solvent A Washes	2
PostInj Solvent B Washes	2
Viscosity Delay	1 seconds
Plunger Speed	Fast

HP5890 Temperature Parameters

Zone Temperatures:	State	Setpoint
Inlet A:	Off	50 C
Inlet B:	On	275 C
Detector A:	Off	50 C
Detector B:	On	300 C
Auxiliary:	Off	50 C

Oven Parameters:

Oven Equib Time:	0.10 minutes
Oven Max:	350 C
Oven State:	On
Cryo State:	Off
Cryo Blast:	Off
Ambient:	25 C

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Oven Program:

Initial Temperature:	60 C
Initial Time:	1.00 minutes

Level	Rate (C/minute)	Final Temperature (C)	Final Time (minutes)
1	35.0	130	0.00
2(A)	12.0	325	4.75
3(B)	0.0	50	1.00

Next Run Time: 24.00 minutes

HP5890 Inlet Pressure Programs

GC Pressure Units: psi

Inlet A:

Constant Flow:	Off
Constant Flow Pressure:	0.0 psi
Constant Flow Temperature:	50 C
Initial Pressure:	0.0 psi
Initial Time:	650.00 minutes

Level	Rate (psi/minute)	Final Pressure (psi)	Final Time (minutes)
1	0.00	0.0	0.00
2(A)	0.00	0.0	0.00
3(B)	0.00	0.0	0.00

Total Program Time: 650.00 minutes

Column Length:	30.00 m
Column Diameter:	0.530 mm
Gas:	He
Vacuum Compensation:	Off

Inlet B:

Constant Flow:	Off
Constant Flow Pressure:	0.0 psi
Constant Flow Temperature:	50 C
Initial Pressure:	7.0 psi
Initial Time:	0.00 minutes

Level	Rate (psi/minute)	Final Pressure (psi)	Final Time (minutes)
1	99.00	36.0	0.50
2(A)	99.00	7.0	0.00
3(B)	0.00	0.0	0.00

Total Program Time: 1.09 minutes

Column Length:	30.00 m
Column Diameter:	0.250 mm
Gas:	He
Vacuum Compensation:	On

HP5890 Packed Column Flow Control

MASTER

Inlet A not used to control packed column flow.

Inlet B not used to control packed column flow.

HP5890 Purge Valve Settings

Inlet Purge	Init Value	On Time	Off Time	Splitless Injection

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A	Off	0.17	0.00	No
B	On	0.17	0.00	No

HP5890 Valve and Relay Information

Initial Setpoints:
5890 Valves:
Valve 1: Off Valve 2: Off Valve 3: Off Valve 4: On
19405 Valves:
Valve 5: Off Valve 6: Off Valve 7: Off Valve 8: Off
19405 Relays:
Relay 1: Off Relay 2: Off Relay 3: Off Relay 4: Off

HP5890 Detector Information

Detector	Type	State
A	---	Off
B	---	Off

HP5890 Signal Information

Not saving signal data.

Signal	Source	Peak Width	Data Rate	Start Data	Stop Data
1	Comp1	0.053	5.000	0.00	1.00
2	Testplot	0.053	5.000	0.00	1.00

MS ACQUISITION PARAMETERS

General Information
------- -----------

Tune File : DFTPP.U
Acquistion Mode : Scan

MS Information
-- -----------

Solvent Delay : 1.20 min

EM Absolute : False
EM Offset : 0
Resulting EM Voltage : 2788.2

[Scan Parameters]

MASTER

Low Mass : 35
High Mass : 500
Threshold : 500
Sample # : 2 A/D Samples 4
Plot 2 low mass : 50
Plot 2 high mass : 550

END OF MS ACQUISITION PARAMETERS

END OF INSTRUMENT CONTROL PARAMETERS

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DATA ANALYSIS PARAMETERS

Method Name: C:\HPCHEM\1\METHODS\PAH2DR.M

Percent Report Settings

Sort By: Signal

Output Destination
    Screen: No
    Printer: Yes
    File: No

Integration Events: Meth Default

Generate Report During Run Method: No

Signal Correlation Window: 0.020

Qualitative Report Settings

Peak Location of Unknown: Apex

Library to Search	Minimum Quality
nbs75k.L	0

Integration Events: Meth Default

Report Type: Summary

Output Destination
    Screen: Yes
    Printer: No
    File: No

Generate Report During Run Method: No

Quantitative Report Settings

Report Type: Summary

Output Destination
    Screen: Yes
    Printer: Yes
    File: No

Generate Report During Run Method: No

MASTER

LL PAH ELEMENT ID 0604005
Calibration Last Updated: Thu Apr 06 15:54:57 2006

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Reference Window: 0.50 Minutes
Non-Reference Window: 0.50 Minutes
Correlation Window: 0.02 minutes
Default Multiplier: 1.00
Default Sample Concentration: 0.00

Compound Information

---

1) 1,4-Dichlorobenzene-d4 (ISTD TR)

Ret. Time 4.21 min., Extract & Integrate from 4.06 to 4.36 min.

Signal	Rel Resp.	Pct. Unc.(rel)	Integration
Tgt 152.00			*** METH DEFAULT ***
Q1 115.00	51.80	30.0	*** METH DEFAULT ***
Q2 150.00	153.70	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ng/uL)	Response
0.2	2.000	5361
0.4	2.000	5216
1.0	2.000	4812
2.0	2.000	10368
5.0	2.000	5671
8.0	2.000	5719
CC	2.000	6400
0.1	2.000	5740

Qualifier Peak Analysis ON ISTD conc: 2.000 ng/uL
Curve Fit: Avg. RF
---

2) 1,2 Dichlorobenzene-d4(SURR) ( )

Ret. Time 4.41 min., Extract & Integrate from 3.91 to 4.91 min.

Signal	Rel Resp.	Pct. Unc.(rel)	Integration
Tgt 152.00			*** METH DEFAULT ***
Q1 150.00	173.80	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ng/uL)	Response
0.2	0.200	588
0.4	0.400	1046
1.0	1.000	2405
2.0	2.000	10416
5.0	5.000	13918
8.0	8.000	23368
CC	1.000	-1
0.1	0.100	384

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

MASTER

3) Naphthalene-d8 (ISTD TR)

Ret. Time 5.67 min., Extract & Integrate from 5.52 to 5.82 min.

Signal	Rel Resp.	Pct. Unc.(rel)	Integration
Tgt 136.00			*** METH DEFAULT ***
Q1 68.00	7.10	30.0	*** METH DEFAULT ***

Lvl	Conc (ng/uL)	Response
0.2	2.000	13794
0.4	2.000	14266
1.0	2.000	13382

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Appendix D

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ESS Laboratory (SVOA)
MS1 Sequence Log

Work Orders Reported: _____

Date Analyzed: _____ Analyst: _____

	8270/625	Seq ID	Calibration ID
Soil / Aqueous			

	Other Method:	Seq ID	Calibration ID
Soil / Aqueous			

** Note in comments when other method used.*

Vial	File	Lab Id	Rw	Rpt	Imp By	STD ID/ Comments	Batch ID	Matrix	-RE Ref
	SV1								
	SV1								
	SV1								
	SV1								
	SV1								
	SV1								
	SV1								
	SV1								
	SV1								
	SV1								
	SV1								
	SV1								
	SV1								
	SV1								
	SV1								
	SV1								
	SV1								
	SV1								
	SV1								
	SV1								
	SV1								
	SV1								
	SV1								
	SV1								
	SV1								
	SV1								
	SV1								

ICAL/CCAL Failures, Quads: _____

Comments: _____

MASTER

Tune Method: DFTPP

ICAL Batch ID: BB71420(Aq); BB71421(Soil)

Final Data Review: _____

Signed copy included with each work order.

ISTD ID : _____

Control #60.0044-0701A

Lobgook Review: _____

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Appendix D.

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ESS Laboratory (SVOA)
MS2 Sequence Log

Work Orders Reported: _____

Date Analyzed: _____ Analyst: _____

	8270/625	Seq ID	Calibration ID
Soil / Aqueous			

	Other Method:	Seq ID	Calibration ID
Soil / Aqueous			

** Note in comments when other method used.*

Vial	File	Lab Id	Rw	Rpt	Imp By	STD ID/ Comments	Batch ID	Matrix	-RE Ref
	SV2								
	SV2								
	SV2								
	SV2								
	SV2								
	SV2								
	SV2								
	SV2								
	SV2								
	SV2								
	SV2								
	SV2								
	SV2								
	SV2								
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	SV2								
	SV2								
	SV2								
	SV2								
	SV2								
	SV2								
	SV2								
	SV2								
	SV2								
	SV2								
	SV2								
	SV2								

ICAL/CCAL Failures, Quads: _____

Comments: _____

WASTE WATER LABORATORY

Tune Method: DFTPP

ICAL Batch ID: BB71420(Aq); BB71421(Soil)

Final Data Review: _____

Signed copy included with each work order.

ISTD ID : _____

Control #60.0043-0702A

Lobgook Review: _____

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Attachment D
SOP 60_8270

ESS LABORATORY

GC/MS 1 MAINTENANCE RECORD

Date	Trouble Observed	Service Done	Initials

All maintenance must be recorded.
Column serial #s and new parts must be noted.

Control #60.0021-0501A

Page ____

Reviewed by _____

MASTER

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Table F-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 8260 and 8270)					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specific criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see Section C.1.f).	NA.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification (See Box D-13)					
LOQ establishment and verification (See Box D-14)					
Tuning	Prior to ICAL and at the beginning of each 12-hour period.	Refer to method for specific ion criteria.	Retune instrument and verify. Rerun affected samples.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be accepted without a valid tune.
Breakdown check (DDT Method 8270 only)	At the beginning of each 12-hour period, prior to analysis of samples.	Degradation ≤ 20% for DDT. Benzidine and pentachlorophenol should be present at their normal responses, and should not exceed a tailing factor of 2.	Correct problem then repeat breakdown check.	Flagging criteria are not appropriate.	No samples shall be run until degradation ≤ 20%.

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Table F-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 8260 and 8270) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Minimum five-point initial calibration (ICAL) for all analytes	ICAL prior to sample analysis.	1. Average response factor (RF) for SPCCs: VOCs ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachlorolethane; ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane. SVOCs ≥ 0.050. 2. RSD for RFs for CCCs: VOCs and SVOCs ≤ 30% and one option below: Option 1: RSD for each analyte ≤ 15%; Option 2: linear least squares regression r ≥ 0.995; Option 3: non-linear regression–coefficient of determination (COD) $r^2 \geq$ 0.99 (6 points shall be used for second order, 7 points shall be used for third order).	Correct problem then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed. Calibration may not be forced through the origin.
Second source calibration verification (ICV)	Once after each ICAL.	All project analytes within ± 20% of true value.	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
Retention time window position establishment for each analyte and surrogate	Once per ICAL.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	

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Table F-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 8260 and 8270) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Evaluation of relative retention times (RRT)	With each sample.	RRT of each target analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	Flagging criteria are not appropriate.	Laboratories may update the retention times based on the CCV to account for minor performance fluctuations or after routine system maintenance (such as column clipping). With each sample, the RRT shall be compared with the most recently updated RRT. If the RRT has changed by more than ±0.06 RRT units since the last update, this indicates a significant change in system performance and the laboratory must take appropriate corrective actions as required by the method and rerun the ICAL to reestablish the retention times.
Continuing calibration verification (CCV)	Daily before sample analysis and every 12 hours of analysis time.	1. Average RF for SPCCs: VOCs ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachloroethane; ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane. SVOCs ≥ 0.050. 2. %Difference/Drift for all target compounds and surrogates: VOCs and SVOCs ≤ 20%D (Note: D = difference when using RFs or drift when using least squares regression or non-linear calibration).	DoD project level approval must be obtained for each of the failed analytes or corrective action must be taken. Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since last acceptable CCV.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

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Table F-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 8260 and 8270) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Internal standards verification	Every field sample, standard, and QC sample.	Retention time ± 30 seconds from retention time of the midpoint standard in the ICAL; EICP area within -50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, apply Q-flag to analytes associated with the non-compliant IS. Flagging criteria are not appropriate for failed standards.	Sample results are not acceptable without a valid IS verification.
Method blank	One per preparatory batch.	No analytes detected > ½ RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common laboratory contaminants, no analytes detected > RL (see Box D-1).	Correct problem, then see criteria in Box D-1. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
LCS containing all analytes to be reported, including surrogates	One per preparatory batch.	QC acceptance criteria specified by DoD, if available. Otherwise, use in-house control limits. In-house control limits may not be greater than ± 3 times the standard deviation of the mean LCS recovery. See Box D-3 and Appendix G.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available (see full explanation in Appendix G).	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch per matrix (see Box D-7).	For matrix evaluation, use LCS acceptance criteria specified by DoD, if available. Otherwise, use in-house LCS control limits.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.

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Table F-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 8260 and 8270) (continued)					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix (see Box D-7).	MSD: For matrix evaluation, use LCS acceptance criteria specified by DoD, if available. Otherwise, use in-house LCS control limits. MSD or sample duplicate: RPD ≤ 30% (between MS and MSD or sample and sample duplicate).	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Surrogate spike	All field and QC samples.	QC acceptance criteria specified by DoD, if available. Otherwise, use in-house control limits.	For QC and field samples, correct problem then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Apply Q-flag to all associated analytes if acceptance criteria are not met.	Alternative surrogates are recommended when there is obvious chromatographic interference.
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

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Table II B-1: Specific QC Requirements and Performance Standards for SVOCs (SW-846 8270D) Using WSC-CAM-II B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
Initial Demonstration of Proficiency	Laboratory Analytical Accuracy & Precision	(1) Must be performed prior to using method on samples. (2) Must be performed for each matrix. (3) Must contain all target analytes. (4) Must follow procedure in Section 8.4 of SW-846 8000B.	No	NA	Refer to Section 8.4 of SW-846 8000B and Section 1.1.2 of this protocol.	NA
GC/MS Tunes with DFTPP	Inter-laboratory Consistency & Comparability	(1) Criteria for DFTPP listed in Table 3 of SW-846 8270D (the same criteria must be used for all analyses). (2) Every 12 hours prior to sample analysis. (3) DDT breakdown must be evaluated and must be <20%. (4) Pentachlorophenol and benzidine peak tailing must be evaluated. Peak tailing factor must be <2 for benzidine and pentachlorophenol. NOTE: Pentachlorophenol tailing must be evaluated when analyzing for acid SVOCs and benzidine tailing must be evaluated when analyzing for base-neutral SVOCs. NOTE: Tune must be performed in full scan mode for SIM analyses.	No	NA	Perform instrument/injection port maintenance as necessary; retune instrument.	Suspend all analyses until tuning non-compliance is rectified. Report DDT breakdown and peak tailing factor exceedances in laboratory narrative.
Initial Calibration	Laboratory Analytical Accuracy	(1) Must be analyzed at least once prior to analyzing samples, when initial calibration verification or continuing calibration does not meet the performance standards, and when major instrument maintenance is performed. (2) Minimum of 5 standards (or 6 if non-linear regression used).	No	RF <0.05; affects nondetect results for affected analyte in all samples analyzed under this initial calibration.	(1) Recalibrate if >10% of target analytes exceed %RSD, "r", or "r^2" criteria. (2) If ≤10% of compounds exceed criteria, recalibration is not required as long as %RSD <40, r >0.98, or r^2 >0.98. (3) If recalculated	Sample analysis cannot proceed without a valid initial calibration. Report non-conforming compounds (%RSD >20, r <0.99, r^2 <0.99 or minimum RF not met) in laboratory narrative. If non-linear regression

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Table II B-1: Specific QC Requirements and Performance Standards for SVOCs (SW-846 8270D) Using WSC-CAM-II B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
		(3) Low standard must be ≤RL. (4) %RSD ≤20, r ≥0.99 (linear regression), or r^2 ≥0.99 (non-linear regression) for each target analyte. (5) If %RSD >20, linear or non-linear regression must be used. (6) Minimum RFs as per Table 4 of SW-846 8270D for lowest concentration standard and for average RF. (7) Must contain all target analytes. (8) Calibration must be performed under the same conditions as the samples. (9) If linear or non-linear regression used, verify the RL by recalculating concentrations in lowest calibration standard using the final calibration curve; recoveries must be 70-130%. (10) SIM: Laboratory must monitor a minimum of two ions per analyte (the primary ion or quantitation ion and a minimum of one confirmation ion); this is required for all target analytes, surrogates and internal standards.			concentrations from the lowest calibration standard are outside of 70-130% recovery range, either: * The RL limit must be reported as an estimated value[2], or * The RL must be raised to the concentration of the next highest calibration standard that exhibits acceptable recoveries when recalculated using the final calibration curve.	(i.e., quadratic equation) is used for calibration, this must be noted in the laboratory narrative along with the compounds affected.
Initial Calibration Verification	Laboratory Analytical Accuracy	(1) Immediately after each initial calibration. (2) Concentration level near midpoint of curve. (3) Prepared using standard source different than used for initial calibration. (4) Must contain all target analytes. (5) Percent recoveries must be between 70-130% for target analytes except for "difficult" analytes[(**)] which must	No	NA	Locate source of problem; recalibrate if >10% of all analytes are outside of criteria.	If recovery is outside of 70-130% for any analyte, including "difficult" analytes[(**)], report non-conforming compounds in laboratory narrative.

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Table II B-1: Specific QC Requirements and Performance Standards for SVOCs (SW-846 8270D) Using WSC-CAM-II B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
		exhibit percent recoveries between 40-160%.				
Continuing Calibration	Laboratory Analytical Accuracy	(1) Every 12 hours prior to the analysis of samples. (2) Concentration level near midpoint of curve. (3) Must contain all target analytes. (4) %D must be ≤20 for each target analyte. (5) Minimum RFs as per Table 4 of SW-846 8270D. (6) Area counts of internal standards in continuing calibration must be between 50 – 200% of the area counts in the associated mid-level initial calibration standard.	No	RF <0.05; affects nondetect results for affected analyte in all samples analyzed under this continuing calibration.	(1) Recalibrate if >20% of all target analytes or >15% of analytes from a particular class (base-neutral or acid) exceed %D criteria. (2) If internal standard is outside of criteria, locate source of problem and reanalyze the continuing calibration. (3) If ≤20% of compounds exceed criteria, recalibration is not required as long as %D <40.	Report non-conforming compounds (%D >20 or minimum RF not met) and associated samples in laboratory narrative.
Method Blank	Laboratory Method Sensitivity (contamination evaluation)	(1) Extracted with every batch or every 20 samples, whichever is more frequent. (2) Matrix-specific (e.g., water, soil). (3) Target analytes must be <RL except for common laboratory contaminants (phthalates) which must be <5x the RL.	Yes	NA	(1) If concentration of contaminant in sample is <10x concentration in blank, locate source of contamination; correct problem; reextract and reanalyze method blank and associated samples. (2) No corrective action required if concentration of contaminant in sample is >10x concentration in blank or if contaminant not detected in sample.	(1) If sample re-extraction is not possible, report non-conformance in laboratory narrative. (2) If contamination of method blanks is suspected or present, the laboratory, using a "B" or some other convention, should qualify the sample results. Blank contamination should also be documented in the laboratory narrative. (3) If re-extraction is performed within holding

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Table II B-1: Specific QC Requirements and Performance Standards for SVOCs (SW-846 8270D) Using WSC-CAM-II B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
						time and yields acceptable method blank results, the laboratory may report results of the re-extraction only. (4) If re-extraction is performed outside of holding time, the laboratory must report results of both the initial extraction and re-extraction.
Laboratory Control Sample (LCS)	Laboratory Analytical Accuracy	(1) Extracted with every batch or every 20 samples, whichever is more frequent. (2) Concentration level near midpoint of curve. (3) Must contain all target analytes. (4) Matrix-specific (e.g., water, soil). (5) Percent recoveries must be between 40-140% for the base-neutral compounds and between 30-130% for the acid compounds except for "difficult" analytes[(**)] which must exhibit percent recoveries between 15-140% (6) Must be prepared in a water-miscible solvent (e.g., acetone, methanol).	Yes	Recovery <10%; affects nondetect results for affected analyte in all samples extracted with this LCS.	(1) Locate source of problem; re-extract and re-analyze LCS and associated samples if >10% of all analytes are outside of criteria. (2) If ≤10% of compounds are outside of the acceptance criteria, re-extraction is not required as long as recoveries are >10%. (3) If >10% of compounds are above the acceptance criteria (>140% for base-neutral compounds and >130% for acid compounds), reextraction is not required if affected compounds were not detected in associated samples.	(1) If sample re-extraction is not possible, report non-conformance in laboratory narrative. (2) If recovery is outside of 40-140% for any base-neutral compound or 30-130% for any acid compound, including "difficult" analytes[(**)], report non-conforming compounds in laboratory narrative. (3) If re-extraction is performed within holding time and yields acceptable LCS results, the laboratory may report results of the re-extraction only. (4) If re-extraction is performed outside of

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Table II B-1: Specific QC Requirements and Performance Standards for SVOCs (SW-846 8270D) Using WSC-CAM-II B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
						holding time, the laboratory must report results of both the initial extraction and re-extraction.
LCS Duplicate	Laboratory Analytical Accuracy & Precision	(1) Extracted with every batch or every 20 samples, whichever is more frequent. (2) Concentration level near midpoint of curve. (3) Must contain all target analytes. (4) Matrix-specific (e.g., water, soil). (5) Percent recoveries must be between 40-140% for the base-neutral compounds and between 30-130% for the acid compounds except for "difficult" analytes[(**)] which must exhibit percent recoveries between 15-140%. (6) RPDs must be ≤20 for waters and ≤30 for solids. (7) Must be prepared in a water-miscible solvent (e.g., acetone, methanol).	Yes	Recovery <10%; affects nondetect results for affected analyte in all samples extracted with this LCS.	(1) Locate source of problem; re-extract and re-analyze LCS and associated samples if >10% of all analytes are outside of recovery acceptance criteria. (2) If ≤10% of compounds are outside of the recovery acceptance criteria, re-extraction is not required as long as recoveries are >10%. (3) If >10% of compounds are above the recovery acceptance criteria (>140% for base-neutral compounds and >130% for acid compounds), reextraction is not required if affected compounds were not detected in associated samples.	(1) if sample re-extraction is not possible, report non-conformance in laboratory narrative. (2) If recovery is outside of 40-140% for any base-neutral compound or 30-130% for any acid compound, including "difficult" analytes[(**)] or if RPD is outside of criteria, report non-conforming compounds in laboratory narrative. (3) If re-extraction is performed within holding time and yields acceptable LCS results, the laboratory may report results of the re-extraction only. (4) If re-extraction is performed outside of holding time, the laboratory must report results of both the initial extraction and re-extraction.

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Table II B-1: Specific QC Requirements and Performance Standards for SVOCs (SW-846 8270D) Using WSC-CAM-II B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
MS/MSD	Method Accuracy & Precision in Sample Matrix	(1) Every 20 samples (at discretion of laboratory or at request of data user). (2) Matrix-specific. (3) Concentration level near midpoint of curve. (4) Must contain all target analytes. (5) Percent recoveries between 40 -140% for the base-neutral compounds and between 30-130% for the acid compounds. (6) RPDs ≤20 for waters and ≤30 for solids. (7) Must be prepared in a water-miscible solvent (e.g., acetone, methanol).	Yes ONLY when requested by the data user	Recovery <10%; affects nondetect result for affected analyte in unspiked sample only.	Check LCS; if recoveries are acceptable in LCS, narrate non-conformance.	Note exceedances in laboratory narrative.
Surrogates	Method Accuracy in Sample Matrix	(1) Minimum of 3 base-neutral surrogates and 3 acid surrogates, at retention times across GC run. Recommended base-neutral surrogates: nitrobenzene-d5, 2-fluorobiphenyl, terphenyl-d14 Recommended acid surrogates: phenol-d5, 2-fluorophenol, 2,4,6-tribromophenol NOTE: For SIM analyses, surrogates used must be representative of compound class of target analytes (e.g., use base-neutral surrogates if analyzing for PAHs and use acid surrogates if analyzing for pentachlorophenol). (2) Percent recoveries in solid matrices must be between 30-130% for all surrogates. Percent recoveries in water matrices must be between 30-130% for base-neutral surrogates and 15-110% for acid surrogates.	Yes	Recovery <10%; affects all nondetect SVOC results associated with the surrogate compound class (base-neutral or acid) in affected sample.	If two or more surrogates for any one class (base-neutral or acid) are outside of limits or if any one surrogate recovers at <10%: (1) Re-extract the sample if surrogate recoveries are low. (2) Re-extract the sample if surrogate recoveries are high and associated SVOCs were detected in the sample. Re-extraction is not required if one of the following exceptions applies: (a) If surrogate recoveries are high and target	(1) Report recoveries outside of acceptance limits in laboratory narrative. (2) If re-extraction yields similar surrogate non-conformances, the laboratory must report results of both extractions. (3) If re-extraction is performed within holding time and yields acceptable surrogate recoveries, the laboratory may report results of the re-extraction only. (4) If re-extraction is performed outside of the

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Table II B-1: Specific QC Requirements and Performance Standards for SVOCs (SW-846 8270D) Using WSC-CAM-II B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
					analytes are not detected in sample, re-extraction is not required. (b) Re-extraction is not required if obvious interference present (e.g., UCM). If obvious interference is present and surrogate recovery would cause rejection of data (i.e., <10%), re-analyze sample on dilution. (c) If a surrogate is diluted to a concentration below that of the lowest calibration standard, reextraction and/or reanalysis is not required.	holding time and yields acceptable surrogate recoveries, the laboratory must report results of both extractions. (5) If sample is not re-extracted due to obvious interference, the laboratory must provide the chromatogram in the data report.
Internal Standards	Laboratory Analytical Accuracy and Method Accuracy in Sample Matrix	(1) Minimum of 6 at retention times across GC run. NOTE: For SIM analyses, the number of internal standards used will be dependent on the analytes of interest. Internal standards must elute in close proximity to the analytes of interest. (2) Area counts in samples must be between 50 – 200% of the area counts in the associated continuing calibration standard. (3) Retention times of internal standards must be within ±30 seconds of retention times in associated continuing calibration standard.	No	Recovery <20%; affects all nondetect results quantitated using affected internal standard in associated sample.	If one or more internal standards are outside of limits, reanalyze sample unless obvious interference present (e.g., UCM). NOTE: If obvious interference is present and internal standard area would cause rejection of data (i.e., <20%), reanalyze sample on dilution.	(1) Report nonconformances in laboratory narrative. Include actual recovery of internal standard and provide summary of analytes quantitated using the internal standard. (2) If reanalysis yields similar internal standard non-conformances, the laboratory must report results of both analyses. (3) If reanalysis is performed within holding

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Table II B-1: Specific QC Requirements and Performance Standards for SVOCs (SW-846 8270D) Using WSC-CAM-II B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
						time and yields acceptable internal standard recoveries, the laboratory may report results of the reanalysis only. (4) If reanalysis is performed outside of the holding time and yields acceptable internal standard recoveries, the laboratory must report results of both analyses. (5) If sample is not reanalyzed due to obvious interference, the laboratory must provide the chromatogram in the data report.
Quantitation	NA	(1) Quantitation must be based on internal standard calibration. (2) The laboratory must use the average response factor, linear or non-linear regression curve generated from the associated initial calibration for quantitation of each analyte. (3) The internal standard used for quantitation must be the one nearest the retention time of the subject analyte. (4) Results must be reported with 2 or more "significant figures" if ≥RL. If reporting values below the RL, report with 1 or more "significant figures".[3]	NA	NA	NA	NA

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Table II B-1: Specific QC Requirements and Performance Standards for SVOCs (SW-846 8270D) Using WSC-CAM-II B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
Identification	NA	Refer to SW-846 8270D, Section 11.6.	NA	NA	NA	NA
General Reporting Issues	NA	(1) The laboratory must only report values ≥ the sample-specific reporting limit; optionally, values below the sample-specific reporting limit can be reported as estimated, if requested. The laboratory must report results for samples and blanks in a consistent manner. (2) Dilutions: If diluted and undiluted analyses are performed, the laboratory should report results for the lowest dilution within the valid calibration range for each analyte. The associated QC (e.g., method blanks, surrogates, etc.) for each analysis must be reported. (3) Refer to Section 3.3, TICs by GC/MS, for guidance. (4) Results for soils/sediments must be reported on a dry-weight basis for comparison to MCP regulatory standards. (5) Refer to Appendix II B-1 for chain-of-custody requirements regarding preservation, cooler temperature, and holding times.	NA	NA	NA	(1) Qualification of the data is required if reporting values below the sample-specific reporting limit. (2) Complete analytical documentation for diluted and undiluted analyses must be made available for review during an audit. (3) TICs will be evaluated at the discretion of the data user consistent with the guidelines presented in Appendix II B–3. (4) The performance of dilutions must be documented in the laboratory narrative or on the report form. Unless due to elevated concentrations of target compounds, reasons for dilutions must be explained in the laboratory narrative. (5) If samples are not preserved properly or are not received with an acceptable cooler temperature, note the non-conformances in the

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Table II B-1: Specific QC Requirements and Performance Standards for SVOCs (SW-846 8270D) Using WSC-CAM-II B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
						laboratory narrative. (6) If samples are extracted and/or analyzed outside of the holding time, note the non-conformances in the laboratory narrative.

** Potentially "difficult" analytes include: 4-chloroaniline, 4-nitrophenol, phenol, 2,4-dinitrophenol

[1]As per Appendix IV of MassDEP Policy #WSC-07-350, *MCP Representativeness Evaluations and Data Usability Assessments*, September 2007, if these results are observed, data users should consider nondetect results as unusable and positive results as estimated with a significant low bias.

[2]If the RL is estimated due to unacceptable recovery of the lowest standard, the CAM RL has not been achieved; Question G of the "MassDEP MCP Analytical Protocol Certification Form" must be answered "NO" and this must be addressed in the laboratory narrative.

[3]Reporting protocol for "significant figures" is a policy decision included for standardization and consistency for reporting of results and is not a definition of "significant" in the scientific or mathematical sense.

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CPSC Phthalate Analysis:

Application: Samples that are prepared and extracted according to the SOP 50_CPSC_Phthalates are analyzed using this procedure. This procedure describes standards, instrument operating conditions, calculations and reporting.

Standards:

Internal Standard: Acenaphthene-D10 (Purchased fro Sigma Aldrich at 200ppm) 10ul of this standard is added to all samples and standards (10 ul for every ml) for a final concentration of 2ppm.

Dibutyl Phthalate ($C_{16}H_{22}O_4$, DBP), Single Component, CAS No. 84-74-2. Analytical grade

Di-(2-ethylhexyl) phthalate ($C_{24}H_{38}O_4$, DEHP), Single Component, CAS No. 117-81-7. Analytical grade

Benzyl Butyl Phthalate ($C_{19}H_{20}O_4$, BBP), Single Component, CAS No. 85-68-7. Analytical grade

Di-n-octyl phthalate ($C_{24}H_{38}O_4$, DnOP), Single Component , CAS No. 117-84-0. Analytical grade

Diisononyl phthalate ($C_{26}H_{42}O_4$, DINP), Multi Component, CAS No. 28553-12-0/68515-48-0. Analytical grade

Diisodecyl phthalate ($C_{28}H_{46}O_4$, DIDP), Multi Component , CAS No. 26761-40-0/68515-49-1. Analytical grade

Calibration standards are prepared as follows:

Single component analytes: These analytes are in the 8270 Low Level Stock solution at 20ppm. Serial dilutions are made for the calibration standards at comcentrations of 0.05, 0.1,0.5, 1.0, 2.0, and 5.0ppm.

Multi Component analytes. A separate standard is prepared for each multi-component analyte at 10,000ppm(0.5g/50mls). These are diluted to create calibration standards at 1,5,10,50 and 100ppm.

Blank Spikes: Prepared by adding 0.5mls of the SVOA MS spiking solution. At a final volume of 50ml this will produce on column concentrations at 1ppm.

CRM (Certified Reference Material) : Use the second source standard from the SVOA 8270 method. Diluted the 20ppm stock standard (1:20) for the 1ppm CRM standard. Add the appropriate amount of IS.

GC-MS Operating Procedures and Quality Control Measures
A GC-MS system with an automatic injector is suggested for the sample analysis.

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The following GC conditions are used (Table 1):

Table 1. GC Conditions Column	HP-5MS; 30m x .22 mm ID x 0.25 µm
Initial Flow Mode	1 ml/min, constant flow (He gas)
Injection Mode	Pulsed splitless
Injection Amount	1 µl
Injection Port Temp	275° C
Pulse Pressure & Time	35 psi, 0.5 min
Purge Flow & Time	20 ml/min, 2 min
Solvent Delay	5 minutes
Initial Oven Temp, Hold Time	50° C, 1 min
Ramp Temp 1, Plateau	30° C/min, 280° C
Ramp Temp 2, Plateau	15° C/min, 310° C
Final Hold Time	4 minutes

Samples are analyzed using the Select Ion Monitoring (SIM) program listed in Table 2. Scan for corresponding ions of each compound listed in a particular stage (e.g., set SIM Stage 3 to scan for 149, 167, 261, 279, 293, and 307 *m/z*). Note that retention times and optimal *m/z* scan values may differ between instruments. For many instruments it will be possible to program the detector to scan for the various ions in the different stages within a single run.

Table 2. SIM Settings **Retention Time (min)**	**Corresponding Ions (*m/z*)**
Scan Stage 1:	*5 - 9.5 minutes*
Acenaphthene-D10 (Internal Standard)	7.93 – 7.99	**188**
DBP	8.52 – 8.57	**149**, *167, 205, 223*
Scan Stage 2:	*9.5 - 10.8 minutes*
BBP	9.84 – 9.93	*91.1,* ***149****, 206*
DEHP	10.42 – 10.49	***149****, 167, 279*
Scan Stage 3:	*10.8 – End*
DnOP	11.15 – 11.24	***149****, 167, 261, 279*
DINP	10.90 – 12.1	***149****, 167, 293*
DIDP	11.20 – 13.00	***149****, 167, 307*

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Analysis

1. Prepare at least four calibration standards for each of the six phthalates of interest (in the range of 0.5 to 10 µg/ml), along with one calibration blank (methylene chloride). Each calibration standard should have an internal standard concentration of 2 µg/ml.
2. Prior to analysis run the 8270 TUNE standard. Spectral ratios must be within criteria to ensure the MS is functioning properly.
3. Analyze standards and blank with the GC-MS. Qualitatively analyze the results to ensure proper retention times and no contamination.
4. Integrate the peak area from valley to valley (the time range listed in Table 2) for each standard. Compounds scanned in Stages 1 ,2 and 3 are integrated using their quantitative ions (in **bold**).
5. Construct a calibration curve from normalized phthalate signals. Normalization is performed by dividing the integrated phthalate signal area by the integrated internal standard signal area.
6. Analyze a CRM to ensure a proper calibration. The analyzed value should be within ±10% of the expected value. If not, prepare new standards and re-run calibration.
7. Analyze the QC standards and all samples. Analyze a CRM if time has passed since the last calibration check. Add the appropriate amount of IS to samples and QC.
8. Quantitate results. If the results are out of the calibration range, return to step 5 of the phthalate extraction method (perform another dilution to get results in calibration range).

Calculations and Results

Results can be reported as follows:

Percentage [Phthalate] = % Phthalate (w/w) = [(C x V x D) / (W x 1000)] x 100

Where

C = Concentration of phthalate in GC-MS sample (in µg/ml)

V = Total volume of THF and hexanes added from steps 2 and 3 of phthalate extraction method

D = Dilution factor from step 5 of phthalate extraction method

W = Weight of sample collected (in mg)

Repeat calculation for each phthalate present in sample

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1,4-Dioxane Analysis using Isotope Dilution

Application: Samples are prepared and extracted in accordance with SW-846 methods 3510 or 3520 and analyzed using modified SW-846 method 8270 GC/MS-SIM. 1,4-Dioxane-d8 is added to the samples prior to extraction. This isotopically labeled compound functions both as internal standard and as surrogate for the target analyte and serves to correct the variability associated with extraction of the target analyte using the specified extraction procedure. Macro preparation methods cited above may be adjusted for micro quantities per SW846 Chapter Two providing QC acceptance criteria are met.

Standards:

Internal Standard / Surrogate: 1,4-Dioxane-d8 (purchased from Absolute Standards, Cat. # 92785, @10,000 ppm), diluted to 1000 ppm in methylene chloride.

Procedure:

GC-MS Operating Procedures and Quality Control Measures

A GC-MS system with an automatic injector is suggested for the sample analysis. The following GC conditions are used (Table 1):

Table 1. GC Conditions Column	HP-5MS; 30m x .22 mm ID x 0.25 µm
Initial Flow Mode	1 ml/min, constant flow (He gas)
Injection Mode	Pulsed splitless
Injection Amount	1 µl
Injection Port Temp	275° C
Pulse Pressure & Time	35 psi, 0.5 min
Purge Flow & Time	20 ml/min, 2 min
Solvent Delay	2 minutes
Initial Oven Temp, Hold Time	50° C, 1 min
Ramp Temp 1, Plateau	30° C/min, 280° C
Ramp Temp 2, Plateau	15° C/min, 310° C
Final Hold Time	4 minutes

Samples are analyzed using Select Ion Monitoring (SIM) scanning, where m/z = 64, 96 for 1,4-Dioxane-d8 and m/z = 58, 88 for 1,4-Dioxane.

Note that retention times and optimal *m/z* scan values may differ between instruments.

Alternative Extraction Procedure: Micro-extraction by 3580A

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35 mL of sample are placed into a 40 mL VOA vial and spiked with 17.5 uL of a 1000 ppm solution of 1,4 Dioxane-d8. For QC, use DI water. Spike blank spikes and matrix spike samples with 175 uL of a 100 ppm solution of 1,4-Dioxane in Acetone.
Add approximately 6 g of muffled Sodium Sulfate and exactly 3.5 mL of Methylene Chloride. Cap the vial tightly and shake vigorously for at least 2 minutes.
Centrifuge the samples first and then turn them upside down and let them settle for a few minutes (the centrifugation keeps the insoluble residue of Sodium Sulfate from caking together on the septum). Then, carefully remove 1.0 mL of the extract through the septum, using a syringe. Spike with regular 8270 IS solution at 20 ppm and analyze.

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ESS Laboratory
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SOP NO. 60_RSK175

METHANE, ETHANE AND ETHENE GASES IN WATER SAMPLES BY GAS CHROMATOGRAPHY ANALYSIS OF SAMPLE HEADSPACE

APPROVED BY:

Operations Manager	Date 6/9/08
QA Officer	Date 6/9/08
Laboratory Director	Date 6/9/08

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METHANE, ETHANE AND ETHENE BY HEADSPACE ANALYSIS

1.0 SCOPE AND APPLICATION

This procedure is applicable to the analysis of parts-per-billion concentrations of dissolved Methane, Ethane and Ethene (Ethylene) in groundwater samples for monitoring natural attenuation. This method may be modified for the analysis of other dissolved gases.

Compound	Mol. Wt.	CAS Number
Methane	16	74-82-8
Ethene (Ethylene)	28	74-85-1
Ethane	30	74-84-0

2.0 SUMMARY OF METHOD

2.1. Water samples are collected in the field in 40 mL VOA vials without headspace. A 4 mL volume of Helium-filled headspace is created in the sample vial by displacing 4 mL of water with high-purity Helium. The sample vial is shaken for 5 minutes prior to analysis and allowed to equilibrate according to the Henry's Law of partial pressures. A 100 µL aliquot of the sample headspace gas is then analyzed by Gas Chromatography, separated on a packed column and detected using a flame ionization detector (FID).

2.2. After quantitation of the gas equilibrated into the prepared headspace, this method permits calculation of the concentration of the dissolved gas in the water before equilibration. Resulting concentrations are expressed as µg/L (ppb) of dissolved gas in water.

3.0 HEALTH AND SAFETY

3.1. Standard precautionary measures used for handling other organic compounds should be sufficient for the safe handling of the analytes targeted by this procedure.

3.2. Each employee has been trained and has acknowledged being trained in the safe use and handling of chemicals being used in the laboratory. This training has been performed according to the ESS Training SOP 80_0016 and by the Chemical Hygiene Plan, SOP No. 90_0001, in conjunction with the Safety orientation.

3.3. All sample and material handling should be done in a hood while using proper protective equipment to minimize exposure to liquid or vapor. Minimum personnel protective equipment includes laboratory safety glasses, a lab coat or apron, and protective gloves.

3.4. The MSDSs for the concentrated chemicals used in the area are kept on file in a central location that is available for the employees for review.

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4.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

Aqueous samples are collected in 40-mL HCl-preserved VOA vials with Silicon-Teflon septa and caps. The samples are stored in a dark walk-in cooler at 4° C. At least three vials should be provided per sample, so samples can be diluted and/or re-analyzed if necessary. ***Samples must be analyzed within 14 days from date sampled.***

5.0 INTERFERENCES

All of the materials used must be demonstrated to be free from interference.

6.0 APPARATUS

6.1. Gas Chromatograph HP5890 II equipped with a flame ionization detector (FID)

6.2. 2.0 m x 0.75 mm Hayesep S micro-packed stainless steel column (Restek)

6.3. Pressurized Helium Cylinder with 2-stage regulator, 2nd stage set @ 10 psi, connected to approximately 3 ft. tubing with a syringe adapter for Luer-lock needles.

6.4. 25 µL, 100 µL, 500 µL and 5 mL gastight syringes.

6.5. 40 mL VOA vials

6.6 **Data system**:

6.6.1 **Computer:** The Semi-Volatiles laboratory has one GC/FID system analyzing Methane, Ethane, Ethene. SVOA GC8 has an AST computer with a Windows 95 operating system. All computer systems are networked to a Windows 2000 server, which is the destination of all files. A differential back-up is performed nightly and a full back is performed each weekend using Veritas Backup Exec to tapes. As the systems acquires and stores data onto the server, the server becomes full. The data is downloaded and archived onto CDs.

6.6.2 **Software:** HP/Agilent Environmental Chemstation - The software is interfaced to a flame ionization detector and allows the continuous acquisition and storage on machine-readable media of all chromatograms obtained throughout the duration of the instrument program. Current version G1045A version 01.00.

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7.0 REAGENTS AND STANDARDS

7.1 Scotty Certified Gas Standards containing Methane, Ethane and Ethene in Nitrogen at concentrations of 1000 ppmv each and 10,000 ppmv each, respectively.

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7.2 A second source standard other than that described in Sec. 7.1.

8.0 PROCEDURE

8.1 In order to create the headspace in the sample vial, the needle connected to the 10 psi Helium line is inserted through the septum into the sample. At the same time, a 5 mL gastight syringe is also inserted. Then, as the plunger in the syringe is pulled out, 4 mL of sample are removed from the vial and replaced with helium from the cylinder. The vial is then turned upside-down in order to isolate the newly created gas space. NOTE: DO NOT hold the vial upside-down while removing the syringe and Helium supply. The excess pressure in the vial can cause more water to leak out through the openings in the septum. After removal of the Helium supply, let the inside pressure equilibrate very briefly before flipping the vial.
Before chromatographic analysis, the sample vial is shaken thoroughly for one minute and allowed to equilibrate for 15 minutes

8.2 Chromatographic Conditions

The following conditions were optimized for the separation of Methane, Ethene and Ethane in a 5-min chromatographic run. These conditions may be varied, but the same conditions must be used for the analysis of the calibration standards as well as all samples associated with the calibration.

- Column: Hayesep S, micropacked, 2 m x 0.75 mm, from Restek.
- Injector Temperature: 220 °C
- Detector Temperature: 220 °C
- Carrier Gas: Helium @ approx. 9 mL/min
- Oven Temperature: 40°C isothermal during analysis, 150 °C when connected but not in use.
- Injection Volume: 100 µL for samples, 2.5 – 100 µL for standards (see calibration table)
- Run time: 5.0 min

Under the above conditions, Methane elutes @ ~0.55 min, Ethene @ ~1.80 min and Ethane @ ~2.50 min.

8.3 Initial Calibration

Appropriate gas mixtures of the three target analytes are commercially available @ 1000 ppmv and 10000 ppmv concentrations. In order to create the gas concentrations used for initial calibration, the injection volume is varied from 2.5 µL to 100 µL, 2.5 µL being the smallest volume that can be reproducibly injected with a gastight syringe. The concentrations are calculated for 100 µL injections (the injection volume for the samples) as listed in the following table:

Table 1: Nominal Concentrations in the Initial Calibration in ppmv

Level	Conc. injected (ppmv)	Vol. injected (µL)	Nom. Conc. (ppmv)

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1	1000	2.5	25
2	1000	10	100
3	1000	25	250
4	10000	10	1000
5	10000	25	2500
6	10000	100	10000

From the Concentration in ppmv, the concentration in the samples (in µg/L or ppb) can be calculated using the Henry's constants (see equations in section 9):

Table 2: Sample Concentrations in the Initial Calibration in µg/L

	Methane	Ethene	Ethane
Henry's Constant	41300	11400	30200
Level	Sample Concentration in µg/L (ppb)		
1	2.36	6.59	4.79
2	9.42	26.4	19.2
3	23.6	65.9	47.9
4	94.2	264	192
5	236	659	479
6	942	2640	1920

Using the above sample concentrations and injection volumes, a 6-point initial calibration is analyzed. It must pass all acceptance criteria before the analysis of samples can begin. The calibration is calculated using the SAMPLE CONCENTRATIONS, not the headspace concentrations. This very much facilitates the sample quantitation, but is sometimes confusing to the newly trained analyst.

8.4 Initial Calibration Acceptance Criteria

8.4.1 The Relative Standard Deviation (RSD) of the calibration factors from the 6 levels for each of the analytes may not exceed 20 %, if the average response factor is to be used for sample quantitation. For linear regression, a regression coefficient of greater than 0.995 must be achieved. Normally, an FID can easily achieve an RSD < 20% over the concentration range of this initial calibration and therefore it is recommended to use the average response factor for quantitation. Background subtraction of blank values is not permitted.

8.4.2 A second source calibration verification (ICV) must be run immediately following the initial calibration and the criteria listed in Table I must be met before sample analysis may proceed.

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8.5 Continuing Calibration Acceptance Criteria

After the initial calibration and ICV have passed and before samples can be analyzed, the calibration must be verified by analyzing a calibration verification standard (CCV). The percent difference (%D) from the average calibration factor of

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each analyte must be ≤ 15%. The CCV must be run every 12 hours, every 20 samples and also at the end of the analytical sequence.

8.6 Sample Analysis

If all initial and continuing calibration criteria are met, sample analysis can proceed. Samples are prepared as described in Section 8.1 and a 100 µL aliquot of the headspace gas is drawn up through the septum into a 0.1 mL gastight syringe. The gas is injected into the GC and the target analytes are determined. A CCV must be analyzed and passed before sample analysis, then at least every 4 hours (or 20 sample injections, whichever comes first) and after the last sample analysis of the day. If a CCV fails %D criteria, the analysis has to be stopped and corrective action must be taken. When the system is back in control all samples run after the last valid CCV must be re-analyzed.

8.7 Dilutions

If the concentration of one or more target analytes measured in the sample exceeds the linearity range of the initial calibration (> level 6), the sample must be diluted and re-analyzed. The original sample can not be used for this purpose. An aliquot of a fresh 40 mL sample has to be taken and quickly but carefully transferred to a 40 mL vial, filled up with DI water and capped. Then the headspace is prepared as described in section 8.1. If no more fresh sample is available, it is permissible to re-inject the original sample at a lower injection volume.

8.8 Logbooks

Before analysis, all injections must be entered into a GC run log. The entries must contain at least the following information:

- Date (MM/DD/YY)
- Sample ID or ESS Lab ID
- Computer File Number
- Method Name
- Analyst's Initials

9.0 CALCULATIONS

9.1 Density Factor of Analyte in Headspace (*DF*):

$$DF = \frac{MW(g/mole) * 273(°K)}{22.4(L/mole) * T(°K)}$$

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Where: *MW* is the molecular weight and
T is the headspace temperature in °K

9.2 Concentration of Analyte in Headspace (*CHS*):

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$$CHS(\mu g/L) = \frac{C(ppmv) * DF * VHS(mL)}{VWP(mL)}$$

Where: *C (ppmv)* is the nominal concentration of the gas standard (Table 1)
VHS is the volume of the headspace (4 mL) and
VWP is the volume of the water phase (36 mL)

9.3 Concentration of Analyte in Water (*CWP*):

$$CWP(\mu g/L) = \frac{C(ppmv) * 55.5 * MW(g/mole)}{HCon}$$

Where: 1L of water equals 55.5 mole and
HCon = Henry's Constant

9.4 Total Sample Concentration (*CS*):

$$CS(\mu g/L) = CHS(\mu g/L) + CWP(\mu g/L)$$

9.5 Matrix Spike

A spiked sample is prepared by spiking a head-spaced blank or sample with a volume of standard. From the concentration of the gas standard used for spiking *(CSP)* and the volume of gas spiked *(VSP)* the final concentration of analytes and the recovery can be calculated as follows:

9.5.1 Spike Amount *(TSP)* in µg:

$$TSP(\mu g) = \frac{CSP(ppmv) * DF * VSP(mL)}{1000}$$

9.5.2 Concentration of Spike (CSP) in µg/L:

$$CSP(\mu g/L) = \frac{TSP(\mu g) * 1000}{VWP(mL)}$$

9.5.3 Matrix spike recovery (%REC):

$$\%REC = \frac{Cspiked(\mu g/L) - Csample(\mu g/L)}{CSP(\mu g/L)} * 100$$

9.6 Calibration Factor *(CF):*

CF = Peak Area / Mass of Compound Injected

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9.7 Percent Relative Standard Deviation *(%RSD):*

$$\%\ RSD = (SD / Average\ CF) \times 100\%$$

Where: SD = standard deviation

9.8 Percent Difference:

$$\%\ D = ((CFv - CF\ ave) / CF\ ave) * 100\%$$

Where:

CFv = Calibration Factor of the verification standard.

$CF\ ave$ = Mean calibration factor from the initial calibration.

10.0 QUALITY ASSURANCE/QUALITY CONTROL

10.1 Method Blank

A method blank must be prepared and analyzed before every batch of 20 field samples. The method blank consists of reagent water and is head-spaced by the procedure described in section 8.1. The analysis of the method blank must not show any of the target analytes above the reporting limit.

10.2 Blank Spike / Blank Spike Duplicate

The blank spike consists of reagent water, head-spaced as described in section 8.1 and then spiked with 200 µL of 10,000 ppmv gas standard and then shaken and equilibrated. According to the equations in section 9.5, the analyte concentration in the blank spike is:

	Methane	Ethene	Ethane
Concentration (µg/L)	36.4	63.6	68.2

A blank spike and a duplicate must be analyzed after the method blank, before each batch of 20 field samples. Recovery for all three analytes must be within 60-140%. RPD should not be more that 25%. If the blank spike fails the criteria, sample analysis cannot commence until the reason for failure is established and corrective action is taken.

10.3 Matrix Spike / Matrix Spike Duplicate

If required by the client, an MS/MSD can be performed, provided that a sufficient number of sample aliquots were received. Two aliquots of a field sample are each head-spaced and then spiked with 200 µL of 10,000 ppmv gas standard, similar to the blank spike. The spiking concentrations and QC limits are the same as in section 10.2. Failure to meet the QC limits indicates matrix interference and is noted in the case narrative.

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10.4 Accuracy and Precision

All analysts performing this method must demonstrate initial proficiency. This is accomplished by generating data of acceptable accuracy and precision for target analytes in a clean matrix. Four blank spikes are prepared as described in section 10.2 and analyzed in a valid analytical sequence. The blank spikes must meet all QC criteria and the RSD of the recoveries must not be greater than 25%. The initial proficiency results are reported as follows and become part of each employee's training file.

10.4.1 **Accuracy Calculation**: Accuracy is defined as the closeness of agreement between an observed value and an accepted reference value. Each of the four spiked samples will be calculated for percent recovery. The average of the percent recovery values is the accuracy result.

10.4.2 **Precision Calculation**: Precision is defined as the agreement of a set of replicate measurements without assumption of knowledge of the true value. Precision is estimated by the relative standard deviation (RSD) of the four QC samples.

$$\%RSD = (s/x)*100\ \%$$

Where: s = Standard Deviation of a finite number of values. On a scientific calculator use the **σxn-1** key.
x = the average of the four QC sample % recoveries.

Reporting Accuracy and Precision: Report Accuracy and Precision data for RSK-175 with the following minimum info:

Analysis Method: Samples Prepared by:
Date Analyzed: Samples Analyzed by:

Target	*% Rec. QC 1*	*% Rec. QC 2*	*% Rec. QC 3*	*% Rec. QC 4*	*Avg. %Rec.*	*Std. Dev.*	*RSD %*

10.5 Method Detections Limits

Method Detection limits are calculated annually from seven consecutive injections of the Level 1 Standard (25 ppmv), run in a valid analytical sequence, as described in SOP 110.0013.

11.0 DATA VALIDATION

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Data validation will be accomplished by reviewing all of the quality control parameters and assuring that they are within recommended ranges and completing the Data Review Checklist.

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11.1 For matrix spikes, the % Recovery should be 60-140%. If the matrix spike is outside limits, check the LCS. If the LCS is within limits, matrix interferences are present and should be noted in the narrative.

11.2 For MS/MSD, the RPD should be +50%. However, there are cases where duplicates may not work. If this is the case, inform client in narrative concerning sample non-homogeneity.

11.3 Analytical batches with Method blanks above the MRL will be re-prepped and re-analyzed with the following exceptions:

- Samples which are at least twenty times higher than the method blank may be reported.
- When the method blank is less than 5% of the regulatory limit associated with the analyte the method blank would be acceptable.
- If the analyte is found in the method blank above the MRL but is not in any of the associated samples, no corrective action is needed.
- Any results that are reported with method blank contamination must be B-flagged.

11.4 For the LCS, the %Recovery should be 60-140. If the LCS is outside this criterion, the analytical batch will be re-analyzed with the following exceptions:

- For LCSs >140%, samples with results below the MRL may be reported. It has been shown that the results above MRL would have been detected.
- For LCSs < 60% (re-extract for USACE/NavyDoD samples), samples with results above a regulatory limit may be reported.
- In some instances there may be insufficient sample to re-extract. The results are to be reported as estimated values when this occurs.
- Any samples that are reported with invalid LCS data must have a notation in the case narrative.

11.5 All unusual observations and method deviations will be noted in the narrative accompanying the data report presented to the client.

11.6 All data is reviewed for accuracy by a second analyst. Results of this review are noted on the data validation checklist in the second level review column and in the comment section.

12.0 REFERENCES

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12.1 SW846 Method 3810, September 1986

12.2 RSKSOP-175: Sample Preparation and Calculations for Dissolved Gas Analysis in Water Samples using a GC Headspace Equilibration Technique.

12.3 New England EPA Regional Website: Technical Guidance for the Natural

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Attenuation Indicators: Methane, Ethane and Ethene, Revision 1, 02/21/02

12.4 Analysis of Dissolved Methane, Ethane and Ethylene in Ground Water by a Standard Gas Chromatographic Technique. Kampbell, D. H. and Vandergrift, Steve A., Journal of Chromatographic Science, Vol. 36, May 1998.

12.5 DOD QSM Final Version 4.1, April 2009

13.0 POLLUTION PREVENTION and WASTE MANAGEMENT

ESS Laboratory's policies on pollution prevention and waste management are covered in SOP 90_0002, Hazardous Waste Contingency and Emergency Response Plan. All employees are trained in the requirements of the SOP.

14.0 DEFINITIONS

Refer to DoD QSM Appendix B – Glossary, which may be found in Network Directory Q:\DOD\ DoDV4.1.

14.1 **Accuracy**: The closeness of agreement between an observed value and an accepted reference value. When applied to a set of observed values, accuracy will be a combination of a random component and of a common systematic error (or bias) component.

14.2 **Batch:** A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 20, then each group of 20 samples or less will all be handled as a separate batch.

14.3 **Bias:** The deviation due to matrix effects of the measured value (xs - xu) from a known spiked amount, where xs is the spiked sample and xu is the un-spiked sample. Bias can be assessed by comparing a measured value to an accepted reference value in a sample of known concentration or by determining the recovery of a known amount of contaminant spiked into a sample (matrix spike).

14.4 **Control Sample:** A QC sample introduced into a process to monitor the performance of the system.

14.5 **Equipment Blank:** A sample of analyte-free media which has been used to rinse the sampling equipment. It is collected after completion of decontamination and prior to sampling. This blank is useful in documenting adequate decontamination of sampling equipment.

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14.6 **Method Reporting Limit:** The lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The MRL is generally 5 to 10 times the MDL. ESS Laboratory sets the MRL to the lowest non-zero standard in the calibration curve or higher.

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14.7 **Field Duplicates:** Independent samples which are collected as close as possible to the same point in space and time. They are two separate samples taken from the same source, stored in separate containers, and analyzed independently. These duplicates are useful in documenting the precision of the sampling process.

14.8 **Blank Spike (BS):** A known matrix spiked with compound(s) representative of the target analytes. This is used to document laboratory performance.

14.9 **Matrix:** The component or substrate (e.g., surface water, drinking water) which contains the analyte of interest.

14.10 **Matrix Duplicate:** An intra-laboratory split sample which is used to document the precision of a method in a given sample matrix.

14.11 **Matrix Spike:** An aliquot of sample spiked with a known concentration of target analyte(s). The spiking occurs prior to sample preparation and analysis. A matrix spike is used to document the bias of a method in a given sample matrix.

14.12 **Matrix Spike Duplicates:** Intra-laboratory split samples spiked with identical concentrations of target analyte(s). The spiking occurs prior to sample preparation and analysis. They are used to document the precision and bias of a method in a given sample matrix.

14.13 **Method Blank:** An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank should be carried through the complete sample preparation and analytical procedure. The method blank is used to document contamination resulting from the analytical process.

14.14 **Method Detection Limit (MDL):** The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix type containing the analyte. See SOP 110_0013 for further explanation.

14.15 **Organic-Free Reagent Water:** For volatiles, all references to water in the method refer to water in which an interferant is not observed at the method detection limit of the compounds of interest. A water purification system is used to generate organic-free de-ionized water.

14.16 **Records:** Include all logbooks, papers, machine readable materials, or other documentary materials, regardless of physical form or characteristics.

14.17 **Surrogate:** An organic compound which is similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which is not normally found in environmental samples.

14.18 **Trip Blank:** A sample of analyte-free media taken from the laboratory to the sampling site and returned to the laboratory unopened. A trip blank is used to

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document contamination attributable to shipping and field handling procedures. This type of blank is useful in documenting contamination of volatile organics samples.

15.0 PERSONNEL QUALIFICATIONS

15.1 BA/BS Chemistry, Natural & Applied Science or equivalent experience.

15.2 All analysts, before performing any analysis, participate in the ESS Laboratory training program (SOP 80.0016). The training process consists of reading the Standard Operating Procedure, gaining instruction on the procedure from an experienced analyst, and performing the initial demonstration of capability.

16.0 TROUBLESHOOTING

See equipment manufacturer's manual(s).

17.0 DATA MANAGEMENT AND RECORDS

16.1 **Data Management**: ESS Laboratory's utilizes the Promium Element LIMS system as part of its Data Management system. Client sample information is entered into ELEMENT LIMS and analyses are assigned to each sample. The LIMS allows EPA hold times, minimum batch QC requirements, and QC criteria to be assigned to each analysis. Standards can be entered and assigned to QC samples through the LIMS. Once analysis has been performed, data is imported using DataTool avoiding manual errors. In conjunction with Crystal Reports, the ELEMENT system allows for a wide variety of reporting formats.

16.2 **Records**: The specific retention periods required in the NELAC Standards, EPA-CFR and state and local statutes are followed or exceeded. At a minimum, data records are retained for five years from last use (10 years for drinking water). If there is a question about whether a record should be retained or disposed because no specific requirement could be found, the record is retained until such time as a retention period is specified. Records are stored in specified-labeled locations and are easily retrievable. All raw data associated with testing is also retained including; computer printouts, chromatograms, review forms, and logbooks.

15.0 ATTACHMENTS (Including TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA)

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17.1 Figure 1 – Data Review Checklist

17.2 Table 1 – Summary of Method Quality Objectives.

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Figure. 1

Lab Data Review Check List

Methane, Ethane, Ethene (RSK-175)

Project Number(s):			
Batch Number(s): **Method:**			
Review Item	Yes (x)	No (x)	N/A (x)
A. **Initial Calibration** 1. Does the curve consist of a minimum five Calibration Standards?			
2. Is the low standard at or below the MRL?			
3. Are the Response Factors and Regression Coefficient within QC limits for appropriate analytes?			
B. **Continuing Calibration** 1. Are the Continuing Calibration Verification (CCV) standards run every 20 samples or less, within 12hrs?			
2. Is the % Drift within QC limits for appropriate analytes? (+/- 15%)			
D. **Sample Analysis** 1. Are sample holding times met? (14 days)			
2. Are all samples with concentrations > the highest standard used for initial calibration diluted and reanalyzed?			
3. Are RTs within limit?			
F. **QC Samples** 1. Is the Method Blank run at the desired frequency and is concentration for target analytes < ½ MRL?			
2. Is the Laboratory Control Sample run at the desired frequency and is its percent recovery within QC limits (60-140%)?			
3. Is the Matrix Spike/Matrix Spike Duplicate run at the desired frequency and is the percent recovery /RPD within QC limits (60-140% RPD50%)?			
G. **Other** 1. Are all non-conformances included and noted?			
2. Are all calculations checked at the minimum frequency?			
3. Did analyst sign/date the appropriate printouts and report sheets?			
4. Are all sample IDs and units checked for transcription errors?			
5. Are all manual integrations checked by a second reviewer to verify why they were performed?			

Comments on any "No" response:

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Analyst:_____ Date:_____ 2nd Level Review:_____ Date: _____

Control Number: 60_RSK-175 5/12/2008

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Table 1
Summary of Method Quality Objectives for RSK-175
Methane, Ethane, Ethene

QC Element	Frequency	Criteria	Corrective Action
Initial Calibration (ICAL)	Instrument set up. Each time the ICV or CCV cannot meet criteria.	• Minimum of 5 standards and contains all analytes • Low standard ≤ MRL • RSD≤20%, R^2≥0.99 (Do not force through zero for LR). • Single analysis at the reporting limit.	• No allowance. Perform maintenance and recalibrate.
Second Source Calibration Verification (ICV)	Immediately following ICAL	All project analytes within established retention time windows and within ± 20% of the expected value from the ICAL	Correct problem; re-run ICV. If that fails, re-run ICAL. No samples may be run until ICAL has been verified.
CCV	Prior to sample analysis, every 12 hours and every 20 samples and at the end of each analytical sequence.	• Concentration level near midpoint of curve • Percent difference or percent drift must be 85-115%.	• If criteria are exceeded then remake and re-analyze CCV. If second consecutive CCV is within criteria then calibration is verified, otherwise re-calibrate system and re-analyze any sample analyzed after the last valid CCV. ***Exception: If CCV is biased high (concentration greater than upper limit) then any non-detect samples may be reported.***

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Method Blank	One per analytical batch of 20 or fewer samples.	• Matrix specific • Analytes < MRL (< ½ MRL for DoD work)	• Report exceedance in project narrative. • Non-detect samples for that analyte may be reported. • Concentrations that are 20x higher than the method blank may be reported. • Samples reported with a contaminated blank must be "B" flagged. • Re-extract if the above exceptions do not apply. If re-extract is within hold, report just the re-extracted data. If re-extract is outside hold then report both sets of data to client.
Blank spike	One per analytical batch of 20 or fewer samples.	• Level between low and mid-level standard • Must contain all single component analytes • Percent recoveries between 60-140% Laboratories must develop in-house limits that are within above criteria.	• Report exceedance in project narrative. • If LCS exhibits high bias and samples are non-detect, then samples may be reported with no corrective action needed. • Re-analyze if the above exceptions do not apply.
Matrix Spike/ Matrix Spike duplicate	One per analytical batch of 20 or fewer samples, if sufficient sample was provided	• Prepared using the same source as the blank spike • Concentration between low and mid-level standard • Percent recoveries between 60-140%. • RPD should be ≤ 50%	• Check LCS, if recoveries are acceptable then note exceedance in project narrative.
Identification and Quantitation		• Analytes must fall within retention time windows.	

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Title: Total Organic Carbon (Combustion Method) SM5310B (19th Edition) & SW-846 9060A

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1.0 Scope and Application

This SOP describes the laboratory procedure for the determination of total organic carbon (TOC).

1.1 Analytes, Matrix(s), and Reporting Limits

This procedure may be used to determine TOC in non-potable water.

The laboratory's routine reporting limit (RL) for TOC in water is 1.0 mg/L.

2.0 Summary of Method

Organic carbon in sample is converted to carbon dioxide (CO_2) by catalytic combustion and measured directly by an infrared detector. The amount of CO_2 present is directly proportional to the concentration of carbonaceous material in the sample.

- SW-846 Method 9060A Total Organic Carbon, Revision 1, November 2004.
- SM5310B, Combustion-Infrared Method, Standard Methods for the Examination of Water and Wastewater, 19th Edition.

If the laboratory has modified its procedure from the reference methods, a list of modifications will be provided in Section 15.0.

3.0 Definitions

A list of terms and definitions are provided in Appendix A.

4.0 Interferences

Carbonate and bicarbonate represent interferences for this test, and are removed by addition of acid and purging with ultra pure air. This step can result in the loss of volatile organic substances. TOC is, therefore, actually analyzed as NPOC (non-purgeable organic carbon)

This procedure is applicable to homogeneous samples that can be injected into the combustion column by means of a microliter type syringe. The opening of the syringe limits the maximum size of particle that may be included in the sample.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

None

5.2 Primary Materials Used

Table 1 lists those materials used in this procedure that have a serious or significant hazard rating along with the exposure limits and primary hazards associated with that material as identified in the MSDS. The table does not include all materials used in the procedure. A complete list of materials used can be found in section 7.0. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS. Any questions regarding the safe handling of these materials should be directed to the laboratory's Environmental Health and Safety Coordinator.

6.0 Equipment and Supplies

- Total Organic Carbon Analyzer/ Autosampler, Shimadzu TOC 5000A or equivalent.
- Glass Vials, 40 & 6 mL
- Volumetric Flasks, Class A, assorted sizes
- Ultra "zero" grade air (compressed cylinder)
- Autopipette: 1.0 & 5.0 mL sizes, Finpipette or equivalent.
- Parafilm

7.0 Reagents and Standards

7.1 Reagents

- Reagent Water
- Hydrochloric Acid, concentrated: J.T. Baker or equivalent

Hydrochloric Acid, 2N: Add 300 mL of reagent water to a 500mL volumetric flask. Slowly add 21 mL of concentrated Hydrochloric acid to the flask and dilute to volume with reagent water. Assign an expiration date of six months and store the solution at room temperature.

7.2 Standards

Potassium Hydrogen Phthalate: Reagent Grade, J.T. Baker or equivalent

Primary Source Potassium Hydrogen Phthalate Solution (1000 mg/L): Measure 2.128 g of potassium hydrogen phthalate into a 1 L volumetric flask and adjust to volume with reagent water. Assign an expiration date of 6 months and store at a temperature of 4°C (±2). Use this solution for the ICAL and matrix spike.

Second Source Standard (ICV/LCS): Second source standard purchased commercially. (ERA catalog #978: TV=1000 mg/L). Dilute the reference standard with reagent water to a

concentration near the midrange calibration standard (5mL stock standard to 500mL = 10.0 mg/L).

8.0 Sample Collection, Preservation, Shipment and Storage

The laboratory does not perform sample collection so these procedures are not included in this SOP. Sampling requirements may be found in the published reference method.

Listed below are minimum sample size, preservation and holding time requirements:

Matrix	Sample Container	Minimum Sample Size	Preservation[1]	Holding Time[2]	Reference
Water	Amber Glass	250 mL	H_2SO_4 or HCl to pH <2 Chilled to ≤ 6°C	28 days	Published Method(s)

[1] The preservation requirement for waters now specified by the EPA is ≤ 6°C. Temperatures less than 2°C are now acceptable so long as the samples are not frozen.
[2]Holding time is determined from date of collection.

Unless otherwise specified by client or regulatory program, after analysis, samples and extracts are retained for a minimum of 30 days after provision of the project report and then disposed of in accordance with applicable regulations.

9.0 Quality Control

9.1 Sample QC

The laboratory prepares the following quality control samples with each batch of samples.

QC Item	Frequency	Acceptance Criteria
Method Blank (MB)	Every 10 samples	<RL
Laboratory Control Sample (ICV/LCS/CCV)	Every 10 samples	%R (85-115)
Sample Duplicate (DU)	Every 10 samples	RPD (≤ 20)
Matrix Spikes (MS)	Per client request	%R (85-115)

9.2 Instrument QC

The laboratory analyzes the following instrument check standards:

QC Item	Frequency	Acceptance Criteria
Initial Calibration (ICAL)	Initial Method Set-Up, Every 6 months, When CCV Fails	Correlation coefficient: ≥ 0.995

9.2.1 Calibration Curve (ICAL)

Prepare a standard curve initially (during method set-up), after major instrument maintenance such as changing of the column catalyst and when failure of the second source standard indicates re-calibration is necessary.

To prepare the calibration standards add a known volume of the 1000 mg/L primary source potassium hydrogen phthalate (KHP) standard to a known volume of reagent water using the amounts specified in the following table:

Working Calibration Standard Formulation

Calibration Level	Parent Standard (mL)	Reagent Water (mL)	Final Concentration (mg/L)
Blank	0	100	0
Level 1	0.10	99.9	1.0
Level 2	0.30	99.7	3.0
Level 3	0.50	99.5	5.0
Level 4	1.0	99.0	10
Level 5	1.5	98.5	15

Analyze the calibration curve standards as follows:

Turn TOC analyzer on and allow it to warm up before use. Select the TOC button at the bottom of the screen to review the instrument details (operation parameter status). When the combustion column has reached sufficient temperature, “OK” will be displayed next to the furnace temperature.

Fill the 40 mL vials in the center of the autosampler tray with the calibration standards. Fill the first vial with reagent water and each subsequent vial with one of the calibration standards such that:

Vial 1 = Blank
Vial 2 = Level 1
Vial 3 = Level 2
Vial 4 = Level 3
Vial 5 = Level 4
Vial 6 = Level 5

Add 20 drops of 2N hydrochloric acid to each calibration standard to remove any inorganic carbon.

Select “File” then “NEW” to initiate a new sample table. Highlight the first row of the table and select “EDIT”, “INSERT STANDARD”, then “NEW”. After this selection has been made a worksheet with three tabs will appear as follows: “Conditions”, “Curve” and “Data”.

Select the tab for “Conditions” \ and enter the following information:

Title:	Date of Calibration (Example 120103.cal)
Filename:	“Date”.toc (Example 120103.toc)
Method:	Linear Regression without zero shift*
Analysis Type:	NPOC
Range:	1
Sparge Time:	10
Injection Volume:	50 uL or 100uL (The entered value may change when the data tab is selected. Be sure to go back into the “conditions” tab to set this volume correctly.)

Select the tab for “Data” and in the column entitled “Sample Name” enter the name of each standard as STD1-STD6 in rows 1-6, respectively. In the “Sample ID” column, enter the concentration of the standard as it corresponds with sample name as S0 (Blank), S1 (Level 1), S3, S5, S10 and S15. In the “concentration” column, enter in the concentration that corresponds to each standard (0, 1.0, 3.0, 5.0, 10, 15). In the “Vial Location” column, enter S1-S6 (Blank-Level 6) in the row that corresponds to the vial positions along with the concentration of those standards. Select “OK” to return to the filename screen. Enter the filename associated with the calibration and select “OK” to save. Enter the number of injections and maximum number of injections as “4”.

Select “Start” to begin analysis.

After analysis, print and review the calibration curve. The correlation coefficient must be $\geq$ 0.995 for the calibration to be considered acceptable. Repeat calibration if criterion is not met. If criterion is met, establish the new calibration curve as the basis for quantitation. To do so, select “File” and “New”. Highlight the first row and select “Edit”, “Insert Sample”. Highlight “ICV.met” and select “Open”. Select the “NPOC” tab and overwrite the filename of the previous calibration curve with the filename of the new calibration curve. Select “OK” then repeat this step for each standard, blank and sample file.

Place the original hardcopy of the calibration curve in the logbook designated for this purpose and provide a photocopy of the calibration curve to data review.

9.3 Sample Preparation

Allow the samples to warm to ambient temperature then transfer ~6 mL of each sample into a 6 mL autosampler vial. Measure additional aliquots for any sample selected as the sample duplicate. NOTE: Caution should be used if samples have a large amount of sediment that is dispersed within the sample when mixed as this can damage the instrument.

If a matrix spike is requested, prepare the matrix spike by combining 0.1 mL of the 1000 mg/L primary source standard with 20 mL of sample in an empty 40 mL sample vial. This preparation yields a MS concentration of 5.0 mg/L. Transfer ~6 mL of spiked sample from 40 mL vial into a 6 mL autosampler vial.

To prepare the method blank (MB), transfer ~40 mL of reagent water into a 40 mL autosampler vial. Prepare additional aliquots for every 10 samples.

To prepare the LCS, transfer ~40 mL of the second source standard into a 40 mL autosampler vial. Prepare additional aliquots for every 10 samples.

Place the vials for the MB and the LCS in the center of the autosampler tray. Note their labeled position so you can enter this information into the software program.

Place the sample vials in the outer portion of the autosampler tray, beginning with position #1.

An example analytical sequence that includes initial calibration (ICAL) is as follows:

CAL Blank
Level 1 / 1.0mg/L

Level 2 / 3.0mg/L
Level 3 / 5.0mg/L
Level 4 / 10.0mg/L
Level 5 / 15.0mg/L
ICV
LCS
MB
10 Samples
MS
SD
LCS
MB
10 Samples
MS
SD
LCS
MB

NOTE: An ICV and an LCS must be run after every ICAL as two separate samples. Runs with no ICAL can open and close with an LCS and the samples can be linked back to the original ICV using the TALS QC Linking tab. The LCS serves as the independently prepared check standard referenced in the method.

Add 3 drops of 2N hydrochloric acid to each 6 mL autosampler vial and 20 drops 2N hydrochloric acid to each 40 mL autosampler vial to remove any inorganic carbon. Cover the vials with parafilm to prevent bubbling over during sparging.

Open a new sample table by selecting "File" and "New". Highlight Row #1 and select "Edit" and "Insert Control". Select the sample type ICV that is appropriate to the vial position in the tray and select "OK". Repeat for the Method Blank, selecting ICB in the software. Then select "Edit" and "Insert Sample" or "Autogenerate" to add each sample, selecting sample type "Sample". Repeat for each blank, standard, sample. In the sample name column, enter the corresponding sample IDs for each field sample. Enter any applicable dilution factor in the Dilution Column. Enter all applicable vial locations for all rows, selecting "S" positions for ICV and Blanks (Example S8 or S1). Place the autosampler cover over the sample tray.

Save the analytical sequence template with the filename "date.toc" for ICAL runs (Example 120103.toc). Designate a letter after the date if not an ICAL run to correspond to each run performed on that date (Example 110410A.toc or 110410B.toc). Select "Background Monitor" and ensure that the background is reading with a flat line around the 0 mark. When this action is complete, select "Start" to initiate analysis. Make sure "keep instrument on" is selected and press OK.

The TOC software is programmed to analyze each sample in quadruplicate and to calculate the average result. TOC concentrations are determined by comparing the peak height to the calibration curve.

Print summary table and chromatograms from TOC software, "File" menu.

Import the results from the instrument to TALS and review data. Schedule re-analysis for samples with concentrations that exceed the range of calibration appropriately.

NOTE: The laboratory does not subtract the calibration blank absorbance value from all other determined responses. The laboratory includes a calibration blank in the generation of the calibration curve and this response is used in the calibration in lieu of a zero value. In this manner, all values are effectively corrected for the calibration blank response.

10.0 Calculations / Data Reduction

LCS Percent Recovery (%R)

$$\%R = \frac{SR}{SA} \otimes 100$$

Where:
SR= LCS Result
SA= LCS True Value

Precision (RPD)

$$RPD = \frac{(D1 - D2)}{\frac{D1 + D2}{2}} \otimes 100$$

Where:
D1 = Sample Result
D2 = Sample Duplicate Result
D2 = Sample Duplicate Result

10.1 Data Review

10.1.1 Primary Review

Review project documents or comments in TALS to ensure project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Enter the worksheet and batch information in the TALS batch.

Evaluate and QC samples against the acceptance criteria given in Table 2. Perform the recommended corrective action as necessary. If corrective action is not taken or is not successful, initiate a nonconformance memo (NCM) to document the situation.

Set results to primary and secondary as appropriate, check QC linkages then set the TALS batch to first level review.

10.1.2 Secondary Data Review

Review project documents or comments in TALS to ensure project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Verify that the performance criteria for the QC items listed in Table 2 were met. If the results do not fall within the established limits verify the recommended corrective actions were performed. If corrective action was not taken or is unsuccessful, ensure the situation is documented with a nonconformance memo (NCM) and ensure data is qualified accordingly.

Verify analytical results are qualified accordingly and the TALS batch information is complete. Set samples to 2nd level review.

Run the LIMS QC Checker, investigate and correct any problems found. Run and review the deliverable. Fix any problems found then set the method chain to lab complete.

10.2 Data Reporting

The report format, application of data qualifiers and creation of the data deliverable is performed by the LIMS using the formatter and deliverable set by the project manager during log-in.

Records of electronic and hardcopy data are maintained as described in laboratory SOP BR-QA-014.

11.0 Method Performance

11.1 Method Detection Limit Study (MDL)

Perform a method detection limit (MDL) study at initial method set-up following the procedures specified in laboratory SOP BR-QA-005.

11.2 Demonstration of Capabilities (DOC)

Perform a method demonstration of capability at initial set-up and when time there is a significant change in instrumentation or procedure.

Each analyst that performs this procedure must complete an initial demonstration of capability (IDOC) prior to independent analysis of client samples. Each analyst must demonstrate on-going proficiency (ODOC) annually thereafter. DOC procedures are further described in the laboratory's quality system manual (QAM) and in the laboratory SOP for employee training, BR-QA-011.

11.3 Training Requirements

Any employee that performs any portion of the procedure described in this SOP must have documentation in their employee training file that they have read this version of this SOP.

Instrument analysts, prior to independent analysis of client samples, must also have documentation of demonstration of initial proficiency (IDOC) and annual on-going proficiency (ODOC) in their employee training files.

12.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability).

13.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to laboratory SOP BR-LP-001.

14.0 References / Cross-References

- SW-846 Method 9060A, Total Organic Carbon, Revision 1, November 2004.
- SM5310B, Combustion-Infrared Method, Standard Methods for the Examination of Water and Wastewater, 19th Edition.
- Laboratory SOP BR-QA-005
- Laboratory SOP BR-QA-011
- Laboratory SOP BR-LP-011
- Laboratory SOP BR-QA-014
- Laboratory Quality Assurance Manual (QAM)

15.0 Method Modifications

The laboratory procedure is modified from the reference method as follows:

Modification Number	Method Reference	Modification
1	SW-846 9060A	The laboratory does not report the range of the quadruplicate replicates.
2	SW-846 9060A SM5310B	The laboratory does not homogenize the samples in a blender prior to analysis.
3	SM5310B	The laboratory does not repeat sample injections until consecutive peaks are obtained and reproducible to ± 10%. The laboratory does analyze each sample in quadruplicate.
5	SW-846 9060A SM5310B	The laboratory analyzes QC samples at a higher frequency than specified in the reference methods.
6	SM5310B	The laboratory uses hydrochloric acid (HCl) instead of phosphoric acid to remove inorganic carbon. HCl is the acid recommended by the instrument manufacturer of the TOC analyzer.
7	SW-846 9060A SM5310B	The laboratory uses a 3 minute sparge time as recommended by the instrument manufacturer instead of the 10 minute sparge specified in the methods.
8	SM5310B	The laboratory does not subtract the calibration blank

		absorbance value from all other determined responses. The laboratory includes a calibration blank in the generation of the calibration curve and this response is used in the calibration in lieu of a zero value. In this manner, all values are effectively corrected for the calibration blank response.

16.0 Attachments

- Table 1: Primary Materials Used
- Table 2: QC Summary and Recommended Corrective Action

17.0 Revision History

BR-WC-002, Revision 13:

- Title Page: Updated SW846 method reference and approval signagures
- Section 2.0: Updated SW846 method reference
- Section 15.0: Updated SW846 method reference

BR-WC-002r12

- Title Page: Updated approval signatures
- All Section: Added reference to new LIMS (TALS) and changed procedures as needed to correlate with LIMS procedures.

BR-WC-002, r11

- Title Page: Updated approval signatures.
- All Sections: Added SM5310B method reference and procedure.

Table 1: Primary Materials Used

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Sulfuric Acid	Corrosive Oxidizer Dehydra-dator	1 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system.
Hydrochloric Acid	Corrosive	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

Table 2: QC Summary

QC Item	Frequency	Acceptance Criteria	Recommended Corrective Action[1]
ICAL	Initially, ever 6 months, when LCS fails	≥ 0.995	Repeat calibration
Method Blank (MB)	Every 10 samples	<RL	Redigest and reanalyze along with associated samples.
LCS/CCV	Every 10 samples	%R (85-115)	Redigest and reanalyze along with associated samples.
Sample Duplicate (DU)	Per client request	RPD (≤ 20)	Examine project requirements, consult with PM to determine appropriate corrective action; qualify data.
Matrix Spike (MS)	Per client request	%R (85-115)	Evaluate LCS, if LCS within criteria, failure is likely matrix effect; qualify data.

Appendix A: Terms and Definitions

Batch: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria.

Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s) processed simultaneously with and under the same conditions as samples through all steps of the procedure.

Matrix Duplicate (DP): duplicate aliquot of a sample processed and analyzed independently; under the same laboratory conditions; also referred to as Sample Duplicate.

Method Blank (MB): a blank matrix processed simultaneously with and under the same conditions as samples through all steps of the procedure. Also known as the preparation blank (PB).

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical, and/or biological integrity of the sample.

Reporting Limit (RL): the level to which data is reported for a specific test method and/or sample.

Company Confidential & Proprietary

Designation: D 445 – 06

An American National Standard
British Standard 2000: Part 71:1990

Designation: 71/1/97

Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)[1]

This standard is issued under the fixed designation D 445; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope*

1.1 This test method specifies a procedure for the determination of the kinematic viscosity, ν, of liquid petroleum products, both transparent and opaque, by measuring the time for a volume of liquid to flow under gravity through a calibrated glass capillary viscometer. The dynamic viscosity, η, can be obtained by multiplying the kinematic viscosity, ν, by the density, ρ, of the liquid.

NOTE 1—For the measurement of the kinematic viscosity and viscosity of bitumens, see also Test Methods D 2170 and D 2171.

NOTE 2—ISO 3104 corresponds to Test Method D 445.

1.2 The result obtained from this test method is dependent upon the behavior of the sample and is intended for application to liquids for which primarily the shear stress and shear rates are proportional (Newtonian flow behavior). If, however, the viscosity varies significantly with the rate of shear, different results may be obtained from viscometers of different capillary diameters. The procedure and precision values for residual fuel oils, which under some conditions exhibit non-Newtonian behavior, have been included.

1.3 The range of kinematic viscosities covered by this test method is from 0.2 to 300 000 mm^2/s (see Table A1.1) at all temperatures (see 6.3 and 6.4). The precision has only been determined for those materials, kinematic viscosity ranges and temperatures as shown in the footnotes to the precision section.

1.4 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 446 Specifications and Operating Instructions for Glass Capillary Kinematic Viscometers

D 1193 Specification for Reagent Water

D 1217 Test Method for Density and Relative Density (Specific Gravity) of Liquids by Bingham Pycnometer

D 1480 Test Method for Density and Relative Density (Specific Gravity) of Viscous Materials by Bingham Pycnometer

D 1481 Test Method for Density and Relative Density (Specific Gravity) of Viscous Materials by Lipkin Bicapillary Pycnometer

D 2162 Practice for Basic Calibration of Master Viscometers and Viscosity Oil Standards

D 2170 Test Method for Kinematic Viscosity of Asphalts (Bitumens)

D 2171 Test Method for Viscosity of Asphalts by Vacuum Capillary Viscometer

D 6071 Test Method for Low Level Sodium in High Purity Water by Graphite Furnace Atomic Absorption Spectroscopy

D 6074 Guide for Characterizing Hydrocarbon Lubricant Base Oils

D 6617 Practice for Laboratory Bias Detection Using Single Test Result from Standard Material

E 1 Specification for ASTM Liquid-in-Glass Thermometers

E 77 Test Method for Inspection and Verification of Thermometers

2.2 *ISO Standards:*[3]

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.07 on Flow Properties.

Current edition approved May 15, 2006. Published June 2006. Originally approved in 1937. Last previous edition approved in 2004 as D 445–04$^{\epsilon 2}$.

In the IP, this test method is under the jurisdiction of the Standardization Committee.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036.

***A Summary of Changes section appears at the end of this standard.**

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ISO 3104 Petroleum Products—Transparent and Opaque Liquids—Determination of Kinematic Viscosity and Calculation of Dynamic Viscosity
ISO 3105 Glass Capillary Kinematic Viscometers—Specification and Operating Instructions
ISO 3696 Water for Analytical Laboratory Use—Specification and Test Methods
ISO 5725 Accuracy (trueness and precision) of measurement methods and results.
ISO 9000 Quality Management and Quality Assurance Standards—Guidelines for Selection and Use
ISO 17025 General Requirements for the Competence of Testing and Calibration Laboratories

2.3 *NIST Standards:*[4]

NIST Technical Note 1297, Guideline for Evaluating and Expressing the Uncertainty of NIST Measurement Results
NIST GMP 11
NIST Special Publication 819

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *automated viscometer*, *n*—apparatus which, in part or in whole, has mechanized one or more of the procedural steps indicated in Section 11 or 12 without changing the principle or technique of the basic manual apparatus. The essential elements of the apparatus in respect to dimensions, design, and operational characteristics are the same as those of the manual method.

3.1.1.1 *Discussion*—Automated viscometers have the capability to mimic some operation of the test method while reducing or removing the need for manual intervention or interpretation. Apparatus which determine kinematic viscosity by physical techniques that are different than those used in this test method are not considered to be Automated Viscometers.

3.1.2 *density*, *n*—the mass per unit volume of a substance at a given temperature.

3.1.3 *dynamic viscosity*, *n*—the ratio between the applied shear stress and rate of shear of a liquid.

3.1.3.1 *Discussion*—It is sometimes called the coefficient of dynamic viscosity or, simply, viscosity. Thus dynamic viscosity is a measure of the resistance to flow or deformation of a liquid.

3.1.3.2 *Discussion*—The term dynamic viscosity can also be used in a different context to denote a frequency-dependent quantity in which shear stress and shear rate have a sinusodial time dependence.

3.1.4 *kinematic viscosity*, *n*—the resistance to flow of a fluid under gravity.

3.1.4.1 *Discussion*—For gravity flow under a given hydrostatic head, the pressure head of a liquid is proportional to its density, ρ. For any particular viscometer, the time of flow of a fixed volume of fluid is directly proportional to its kinematic viscosity, ν, where $\nu = \eta/\rho$, and η is the dynamic viscosity coefficient.

[4] Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 3460, Gaithersburg, MD 20899-3460.

4. Summary of Test Method

4.1 The time is measured for a fixed volume of liquid to flow under gravity through the capillary of a calibrated viscometer under a reproducible driving head and at a closely controlled and known temperature. The kinematic viscosity (determined value) is the product of the measured flow time and the calibration constant of the viscometer. Two such determinations are needed from which to calculate a kinematic viscosity result that is the average of two acceptable determined values.

5. Significance and Use

5.1 Many petroleum products, and some non-petroleum materials, are used as lubricants, and the correct operation of the equipment depends upon the appropriate viscosity of the liquid being used. In addition, the viscosity of many petroleum fuels is important for the estimation of optimum storage, handling, and operational conditions. Thus, the accurate determination of viscosity is essential to many product specifications.

6. Apparatus

6.1 *Viscometers*—Use only calibrated viscometers of the glass capillary type, capable of being used to determine kinematic viscosity within the limits of the precision given in the precision section.

6.1.1 Viscometers listed in Table A1.1, whose specifications meet those given in Specifications D 446 and in ISO 3105 meet these requirements. It is not intended to restrict this test method to the use of only those viscometers listed in Table A1.1. Annex A1 gives further guidance.

6.1.2 *Automated Viscometers*—Automated apparatus may be used as long as they mimic the physical conditions, operations or processes of the manual apparatus. Any viscometer, temperature measuring device, temperature control, temperature controlled bath or timing device incorporated in the automated apparatus shall conform to the specification for these components as stated in Section 6 of this test method. Flow times of less than 200 s are permitted, however, a kinetic energy correction shall be applied in accordance with Section 7 on Kinematic Viscosity Calculation of Specifications D 446. The kinetic energy correction shall not exceed 3.0 % of the measured viscosity. The automated apparatus shall be capable of determining kinematic viscosity of a certified viscosity reference standard within the limits stated in 9.2.1 and Section 17. The precision shall be of statistical equivalence to, or better (has less variability) than the manual apparatus.

NOTE 3—Precision and bias of kinematic viscosity measurements for flow times of less than 200 s has not been determined. The precision stated in Section 17 is not know to be valid for kinematic viscosity measurements with flow times less than 200 s.

6.2 *Viscometer Holders*—Use viscometer holders to enable all viscometers which have the upper meniscus directly above the lower meniscus to be suspended vertically within 1° in all directions. Those viscometers whose upper meniscus is offset from directly above the lower meniscus shall be suspended vertically within 0.3° in all directions (see Specifications D 446 and ISO 3105).

6.2.1 Viscometers shall be mounted in the constant temperature bath in the same manner as when calibrated and stated on the certificate of calibration. See Specifications D 446, see Operating Instructions in Annexes A1–A3. For those viscometers which have Tube L (see Specifications D 446) held vertical, vertical alignment shall be confirmed by using (*1*) a holder ensured to hold Tube L vertical, or (*2*) a bubble level mounted on a rod designed to fit into Tube L, or (*3*) a plumb line suspended from the center of Tube L, or (*4*) other internal means of support provided in the constant temperature bath.

6.3 *Temperature-Controlled Bath*—Use a transparent liquid bath of sufficient depth such, that at no time during the measurement of flow time, any portion of the sample in the viscometer is less than 20 mm below the surface of the bath liquid or less than 20 mm above the bottom of the bath.

6.3.1 *Temperature Control*—For each series of flow time measurements, the temperature control of the bath liquid shall be such that within the range from 15 to 100°C, the temperature of the bath medium does not vary by more than ±0.02°C of the selected temperature over the length of the viscometer, or between the position of each viscometer, or at the location of the thermometer. For temperatures outside this range, the deviation from the desired temperature must not exceed ±0.05°C.

6.4 *Temperature Measuring Device in the Range from 0 to 100°C*—Use either calibrated liquid-in-glass thermometers (Annex A2) of an accuracy after correction of ±0.02°C or better, or any other thermometric device of equal or better accuracy.

6.4.1 If calibrated liquid-in-glass thermometers are used, the use of two thermometers is recommended. The two thermometers shall agree within 0.04°C.

6.4.2 Outside the range from 0 to 100°C, use either calibrated liquid-in-glass thermometers of an accuracy after correction of ±0.05°C or better, or any other thermometric device of equal or better accuracy. When two temperature measuring devices are used in the same bath, they shall agree within ±0.1°C.

6.4.3 When using liquid-in-glass thermometers, such as those in Table A2.1, use a magnifying device to read the thermometer to the nearest ⅕ division (for example, 0.01°C or 0.02°F) to ensure that the required test temperature and temperature control capabilities are met (see 10.1). It is recommended that thermometer readings (and any corrections supplied on the certificates of calibrations for the thermometers) be recorded on a periodic basis to demonstrate compliance with the test method requirements. This information can be quite useful, especially when investigating issues or causes relating to testing accuracy and precision.

6.5 *Timing Device*—Use any timing device that is capable of taking readings with a discrimination of 0.1 s or better and has an accuracy within ±0.07 % (see Annex A3) of the reading when tested over the minimum and maximum intervals of expected flow times.

6.5.1 Electrical timing devices may be used if the current frequency is controlled to an accuracy of 0.05 % or better. Alternating currents, as provided by some public power systems, are intermittently rather than continuously controlled. When used to actuate electrical timing devices, such control can cause large errors in kinematic viscosity flow time measurements.

7. Reagents and Materials

7.1 *Chromic Acid Cleaning Solution*, or a nonchromium-containing, strongly oxidizing acid cleaning solution. (**Warning—**Chromic acid is a health hazard. It is toxic, a recognized carcinogen, highly corrosive, and potentially hazardous in contact with organic materials. If used, wear a full face-shield and full-length protective clothing including suitable gloves. Avoid breathing vapor. Dispose of used chromic acid carefully as it remains hazardous. Nonchromium-containing, strongly oxidizing acid cleaning solutions are also highly corrosive and potentially hazardous in contact with organic materials, but do not contain chromium which has special disposal problems.)

7.2 *Sample Solvent*, completely miscible with the sample. Filter before use.

7.2.1 For most samples a volatile petroleum spirit or naphtha is suitable. For residual fuels, a prewash with an aromatic solvent such as toluene or xylene may be necessary to remove asphaltenic material.

7.3 *Drying Solvent*, a volatile solvent miscible with the sample solvent (see 7.2) and water (see 7.4). Filter before use.

7.3.1 Acetone is suitable. (**Warning—**Extremely flammable.)

7.4 *Water*, deionized or distilled and conforming to Specification D 1193 or Grade 3 of ISO 3696. Filter before use.

8. Certified Viscosity Reference Standards

8.1 Certified viscosity reference standards shall be certified by a laboratory that has been shown to meet the requirements of ISO 17025 by independent assessment. Viscosity standards shall be traceable to master viscometer procedures described in Test Method D 2162.

8.2 The uncertainty of the certified viscosity reference standard shall be stated for each certified value (k = 2, 95% confidence). See ISO 5725 or NIST 1297.

9. Calibration and Verification

9.1 *Viscometers*—Use only calibrated viscometers, thermometers, and timers as described in Section 6.

9.2 *Certified Viscosity Reference Standards (*Table A1.2*)*—These are for use as confirmatory checks on the procedure in the laboratory.

9.2.1 If the determined kinematic viscosity does not agree within the acceptable tolerance band, as calculated from Annex A4, of the certified value, recheck each step in the procedure, including thermometer and viscometer calibration, to locate the source of error. Annex A1 gives details of standards available.

NOTE 4—In previous issues of Test Method D 445, limits of ±0.35% of the certified value have been used. The data to support the limit of ±0.35% cannot be verified. Annex A4 provides instructions on how to determine the tolerance band. The tolerance band combines both the uncertainty of the certified viscosity reference standard as well as the uncertainty of the laboratory using the certified viscosity reference standard.

9.2.1.1 As an alternative to the calculation in Annex A4, the approximate tolerance bands in Table 1 may be used.

9.2.2 The most common sources of error are caused by particles of dust lodged in the capillary bore and temperature measurement errors. It must be appreciated that a correct result obtained on a standard oil does not preclude the possibility of a counterbalancing combination of the possible sources of error.

9.3 The calibration constant, C, is dependent upon the gravitational acceleration at the place of calibration and this must, therefore, be supplied by the standardization laboratory together with the instrument constant. Where the acceleration of gravity, g, differs by more that 0.1 %, correct the calibration constant as follows:

$$C_2 = (g_2/g_1) \times C_1 \quad (1)$$

where the subscripts 1 and 2 indicate, respectively, the standardization laboratory and the testing laboratory.

10. General Procedure for Kinematic Viscosity

10.1 Adjust and maintain the viscometer bath at the required test temperature within the limits given in 6.3.1 taking account of the conditions given in Annex A2 and of the corrections supplied on the certificates of calibration for the thermometers.

10.1.1 Thermometers shall be held in an upright position under the same conditions of immersion as when calibrated.

10.1.2 In order to obtain the most reliable temperature measurement, it is recommended that two thermometers with valid calibration certificates be used (see 6.4).

10.1.3 They should be viewed with a lens assembly giving approximately five times magnification and be arranged to eliminate parallax errors.

10.2 Select a clean, dry, calibrated viscometer having a range covering the estimated kinematic viscosity (that is, a wide capillary for a very viscous liquid and a narrower capillary for a more fluid liquid). The flow time for manual viscometers shall not be less than 200 s or the longer time noted in Specifications D 446. Flow times of less than 200 s are permitted for automated viscometers, provided they meet the requirements of 6.1.2.

10.2.1 The specific details of operation vary for the different types of viscometers listed in Table A1.1. The operating instructions for the different types of viscometers are given in Specifications D 446.

10.2.2 When the test temperature is below the dew point, fill the viscometer in the normal manner as required in 11.1. To ensure that moisture does not condense or freeze on the walls of the capillary, draw the test portion into the working capillary and timing bulb, place rubber stoppers into the tubes to hold the test portion in place, and insert the viscometer into the bath. After insertion, allow the viscometer to reach bath temperature, and the remove the stoppers. When performing manual viscosity determinations, do not use those viscometers which cannot be removed from the constant temperature bath for charging the sample portion.

10.2.2.1 The use of loosely packed drying tubes affixed to the open ends of the viscometer is permitted, but not required. If used, the drying tubes shall fit the design of the viscometer and not restrict the flow of the sample by pressures created in the instrument.

10.2.3 Viscometers used for silicone fluids, fluorocarbons, and other liquids which are difficult to remove by the use of a cleaning agent, shall be reserved for the exclusive use of those fluids except during their calibration. Subject such viscometers to calibration checks at frequent intervals. The solvent washings from these viscometers shall not be used for the cleaning of other viscometers.

TABLE 1 Approximate Tolerance Bands

NOTE—The tolerance bands were determined using Practice D 6617. The calculation is documented in Research Report RR: D02–1490.[A]

Viscosity of Reference Material, mm^2/s	Tolerance Band
< 10	±0.30%
10 to 100	±0.32%
100 to 1000	±0.36%
1000 to 10 000	±0.42%
10 000 to 100 000	±0.54%
> 100 000	±0.73%

[A]Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02–1490.

11. Procedure for Transparent Liquids

11.1 Charge the viscometer in the manner dictated by the design of the instrument, this operation being in conformity with that employed when the instrument was calibrated. If the sample is thought or known to contain fibers or solid particles, filter through a 75 µm screen, either prior to or during charging (see Specifications D 446).

NOTE 5—To minimize the potential of particles passing through the filter from aggregating, it is recommended that the time lapse between filtering and charging be kept to a minimum.

11.1.1 In general, the viscometers used for transparent liquids are of the type listed in Table A1.1, A and B.

11.1.2 With certain products which exhibit *gel-like* behavior, exercise care that flow time measurements are made at sufficiently high temperatures for such materials to flow freely, so that similar kinematic viscosity results are obtained in viscometers of different capillary diameters.

11.1.3 Allow the charged viscometer to remain in the bath long enough to reach the test temperature. Where one bath is used to accommodate several viscometers, never add or withdraw, or clean a viscometer while any other viscometer is in use for measuring a flow time.

11.1.4 Because this time will vary for different instruments, for different temperatures, and for different kinematic viscosities, establish a safe equilibrium time by trial.

11.1.4.1 Thirty minutes should be sufficient except for the highest kinematic viscosities.

11.1.5 Where the design of the viscometer requires it, adjust the volume of the sample to the mark after the sample has reached temperature equilibrium.

11.2 Use suction (if the sample contains no volatile constituents) or pressure to adjust the head level of the test sample to a position in the capillary arm of the instrument about 7 mm above the first timing mark, unless any other value is stated in the operating instructions for the viscometer. With the sample flowing freely, measure, in seconds to within 0.1 s, the time required for the meniscus to pass from the first to the second

timing mark. If this flow time is less than the specified minimum (see 10.2), select a viscometer with a capillary of smaller diameter and repeat the operation.

11.2.1 Repeat the procedure described in 11.2 to make a second measurement of flow time. Record both measurements.

11.2.2 From the two measurements of flow time, calculate two determined values of kinematic viscosity.

11.2.3 If the two determined values of kinematic viscosity calculated from the flow time measurements agree within the stated determinability figure (see 17.1.1) for the product, use the average of these determined values to calculate the kinematic viscosity result to be reported. Record the result. If not, repeat the measurements of flow times after a thorough cleaning and drying of the viscometers and filtering (where required, see 11.1) of the sample until the calculated kinematic viscosity determinations agree with the stated determinability.

11.2.4 If the material or temperature, or both, is not listed in 17.1.1, for temperatures between 15 and 100ºC, use as an estimate of the determinability 0.20% and 0.35% for temperatures outside this range.

12. Procedure for Opaque Liquids

12.1 For steam-refined cylinder oils and black lubricating oils, proceed to 12.3 ensuring a thoroughly representative sample is used. The kinematic viscosity of residual fuel oils and similar waxy products can be affected by the previous thermal history and the following procedure described in 12.1.1-12.2.2 shall be followed to minimize this.

12.1.1 In general, the viscometers used for opaque liquids are of the reverse-flow type listed in Table A1.1, C.

12.1.2 Heat in the original container, in an oven, at 60 ± 2°C for 1 h.

12.1.3 Thoroughly stir the sample with a suitable rod of sufficient length to reach the bottom of the container. Continue stirring until there is no sludge or wax adhering to the rod.

12.1.4 Recap the container tightly and shake vigorously for 1 min to complete the mixing.

12.1.4.1 With samples of a very waxy nature or oils of high kinematic viscosity, it may be necessary to increase the heating temperature above 60°C to achieve proper mixing. The sample should be sufficiently fluid for ease of stirring and shaking.

12.2 Immediately after completing 12.1.4, pour sufficient sample to fill two viscometers into a 100-mL glass flask and loosely stopper.

12.2.1 Immerse the flask in a bath of boiling water for 30 min. (**Warning—**Exercise care as vigorous boil-over can occur when opaque liquids which contain high levels of water are heated to high temperatures.)

12.2.2 Remove the flask from the bath, stopper tightly, and shake for 60 s.

12.3 Two determinations of the kinematic viscosity of the test material are required. For those viscometers that require a complete cleaning after each flow time measurement, two viscometers may be used. A single viscometer in which an immediate, repeat flow time measurement can be made without cleaning may also be used for the two measurements of flow time and calculation of kinematic viscosity. Charge two viscometers in the manner dictated by the design of the instrument. For example, for the cross-arm or the BS U-tube viscometers for opaque liquids, filter the sample through a 75-µm filter into two viscometers previously placed in the bath. For samples subjected to heat treatment, use a preheated filter to prevent the sample coagulating during the filtration.

12.3.1 Viscometers which are charged before being inserted into the bath may need to be preheated in an oven prior to charging the sample. This is to ensure that the sample will not be cooled below test temperature.

12.3.2 After 10 min, adjust the volume of the sample (where the design of the viscometer requires) to coincide with the filling marks as in the viscometer specifications (see Specifications D 446).

12.3.3 Allow the charged viscometers enough time to reach the test temperature (see 12.3.1). Where one bath is used to accommodate several viscometers, never add or withdraw, or clean a viscometer while any other viscometer is in use for measuring flow time.

12.4 With the sample flowing freely, measure in seconds to within 0.1 s, the time required for the advancing ring of contact to pass from the first timing mark to the second. Record the measurement.

12.4.1 In the case of samples requiring heat treatment described in 12.1 through 12.2.1, complete the measurements of flow time within 1 h of completing 12.2.2. Record the measured flow times.

12.5 Calculate kinematic viscosity, ν, in mm^2/s, from each measured flow time. Regard these as two determined values of kinematic viscosity.

12.5.1 For residual fuel oils, if the two determined values of kinematic viscosity agree within the stated determinability figure (see 17.1.1), use the average of these determined values to calculate the kinematic viscosity result to be reported. Record the result. If the calculated kinematic viscosities do not agree, repeat the measurements of flow times after thorough cleaning and drying of the viscometers and filtering of the sample. If the material or temperature, or both, is not listed in 17.1.1, for temperatures between 15 and 100°C use as an estimate of the determinability 1.0 %, and 1.5 % for temperatures outside this range; it must be realized that these materials can be non-Newtonian, and can contain solids which can come out of solution as the flow time is being measured.

13. Cleaning of Viscometer

13.1 Between successive determinations of kinematic viscosity, clean the viscometer thoroughly by several rinsings with the sample solvent, followed by the drying solvent (see 7.3). Dry the tube by passing a slow stream of filtered dry air through the viscometer for 2 min or until the last trace of solvent is removed.

13.2 Periodically clean the viscometer with the cleaning solution (**Warning**—see 7.1), for several hours to remove residual traces of organic deposits, rinse thoroughly with water (7.4) and drying solvent (see 7.3), and dry with filtered dry air or a vacuum line. Remove any inorganic deposits by hydrochloric acid treatment before the use of cleaning acid, particularly if the presence of barium salts is suspected. (**Warning—**It is essential that alkaline cleaning solutions are not used as changes in the viscometer calibration can occur.)

14. Calculation

14.1 Calculate each of the determined kinematic viscosity values, ν_1 and ν_2, from the measured flow times, t_1 and t_2, and the viscometer constant, C, by means of the following equation:

$$\nu_{1,2} = C \cdot t_{1,2} \quad (2)$$

where:

$\nu_{1,2}$ = determined kinematic viscosity values for ν_1 and ν_2, respectively, mm^2/s,
C = calibration constant of the viscometer, mm^2/s^2, and
$t_{1,2}$ = measured flow times for t_1 and t_2, respectively, s.

Calculate the kinematic viscosity result, ν, as an average of ν_1 and ν_2 (see 11.2.3 and 12.5.1).

14.2 Calculate the dynamic viscosity, η, from the calculated kinematic viscosity, ν, and the density, ρ, by means of the following equation:

$$\eta = \nu \times \rho \times 10^{-3} \quad (3)$$

where:

η = dynamic viscosity, mPa·s,
ρ = density, kg/m^3, at the same temperature used for the determination of the kinematic viscosity, and
ν = kinematic viscosity, mm^2/s.

14.2.1 The density of the sample can be determined at the test temperature of the kinematic viscosity determination by an appropriate method such as Test Methods D 1217, D 1480, or D 1481.

15. Expression of Results

15.1 Report the test results for the kinematic or dynamic viscosity, or both, to four significant figures, together with the test temperature.

16. Report

16.1 Report the following information:

16.1.1 Type and identification of the product tested,

16.1.2 Reference to this test method or a corresponding international standard,

16.1.3 Result of the test (see Section 15),

16.1.4 Any deviation, by agreement or otherwise, from the procedure specified,

16.1.5 Date of the test, and

16.1.6 Name and address of the test laboratory.

17. Precision

17.1 *Comparison of Determined Values*:

17.1.1 *Determinability (d)*—The difference between successive determined values obtained by the same operator in the same laboratory using the same apparatus for a series of operations leading to a single result, would in the long run, in the normal and correct operation of this test method, exceed the values indicated only in one case in twenty:

Base oils at 40 and 100°C[5]	0.0020 y	(0.20 %)
Formulated oils at 40 and 100°C[6]	0.0013 y	(0.13 %)
Formulated oils at 150°C[7]	0.015 y	(1.5 %)
Petroleum wax at 100°C[8]	0.0080 y	(0.80 %)
Residual fuel oils at 80 and 100°C[9]	0.011 (y + 8)	
Residual fuel oils at 50°C[9]	0.017 y	(1.7 %)
Additives at 100°C[10]	$0.00106\ y^{1.1}$	
Gas oils at 40°C[11]	0.0013 (y+1)	
Jet fuels at –20°C[12]	0.0018 y	(0.18 %)

where: y is the average of determined values being compared.

17.2 *Comparison of Results*:

17.2.1 *Repeatability (r)*—The difference between successive results obtained by the same operator in the same laboratory with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of this test method, exceed the values indicated only in one case in twenty:

Base oils at 40 and 100°C[5]	0.0011 x	(0.11 %)
Formulated oils at 40 and 100°C[6]	0.0026 x	(0.26 %)
Formulated oils at 150°C[7]	0.0056 x	(0.56 %)
Petroleum wax at 100°C[8]	$0.0141\ x^{1.2}$	
Residual fuel oils at 80 and 100°C[9]	0.013 (x + 8)	
Residual oils at 50°C[9]	0.015 x	(1.5 %)
Additives at 100°C[10]	$0.00192\ x^{1.1}$	
Gas oils at 40°C[11]	0.0043 (x+1)	
Jet fuels at –20°C[12]	0.007 x	(0.7 %)

where: x is the average of results being compared.

17.2.2 *Reproducibility (R)*—The difference between two single and independent results obtained by different operators working in different laboratories on nominally identical test material would, in the long run, in the normal and correct operation of this test method, exceed the values indicated below only in one case in twenty.

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[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Reports RR:D02-1331 and RR:D02-1132. These precision values were obtained by statistical examination of interlaboratory results from six mineral oils (base oils without additive package) in the range from 8 to 1005 mm^2/s at 40°C and from 2 to 43 mm^2/s at 100°C, and were first published in 1989. Request See Guide D 6074.

[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1332. These precision values were obtained by statistical examination of interlaboratory results from seven fully formulated engine oils in the range from 36 to 340 mm^2/s at 40°C and from 6 to 25 mm^2/s at 100°C, and were first published in 1991. See Guide D 6071.

[7] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1333. These precision values were obtained by statistical examination of interlaboratory results for eight fully formulated engine oils in the range from 7 to 19 mm^2/s at 150°C, and first published in 1991. See Guide D 6074.

[8] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1334. These precision values were obtained by statistical examination of interlaboratory results from five petroleum waxes in the range from 3 to 16 mm^2/s at 100°C, and were first published in 1988.

[9] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1198. These precision values were obtained by statistical examination of interlaboratory results from fourteen residual fuel oils in the range from 30 to 1300 mm^2/s at 50°C and from 5 to 170 mm^2/s at 80 and 100°C, and were first published in 1984.

[10] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1421. These precision values were obtained by statistical examination of interlaboratory results from eight additives in the range from 145 to 1500 mm^2/s at 100°C and were first available in 1997.

[11] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1422. These precision values were obtained by statistical examination of interlaboratory results from eight gas oils in the range from 1 to 13 mm^2/s at 40°C and were first available in 1997.

[12] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1420. These precision values were obtained by statistical examination of interlaboratory results from nine jet fuels in the range from 4.3 to 5.6 mm^2/s at– 20°C and were first available in 1997.

Base oils at 40 and 100°C[5]	0.0065 x	(0.65 %)
Formulated oils at 40 and 100°C[6]	0.0076 x	(0.76 %)
Formulated oils at 150°C[7]	0.018 x	(1.8 %)
Petroleum wax at 100°C[8]	$0.0366\ x^{1.2}$	
Residual fuel oils at 80 and 100°C[9]	0.04 (x + 8)	
Residual oils at 50°C[9]	0.074 x	(7.4 %)
Additives at 100°C[10]	$0.00862\ x^{1.1}$	
Gas oils at 40°C[11]	0.0082 (x+1)	
Jet fuels at –20°C[12]	0.019 x	(1.9 %)

where: x is the average of results being compared.

17.3 The precision for used oils has not been determined but is expected to be poorer than that for formulated oils. Because of the extreme variability of such used oils, it is not anticipated that the precision of used oils will be determined.

17.4 The precision for specific automated viscometers has not been determined. However, an analysis has been made of a large data set including both automated and manual viscometers over the temperature range of 40 to 100°C. The reproducibility of automated viscometer data is not statistically significantly different than the reproducibility of manual viscometer data. It is also shown that there is no bias of the automated data in comparison to the manual data.[13]

18. Keywords

18.1 dynamic viscosity; kinematic viscosity; viscometer; viscosity

[13] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1498.

ANNEXES

(Mandatory Information)

A1. VISCOMETER TYPES AND CERTIFIED VISCOSITY REFERENCE STANDARDS

A1.1 Viscometer Types

A1.1.1 Table A1.1 lists capillary viscometers commonly in use for viscosity determinations on petroleum products. For specifications, operating instructions, and calibration, refer to specifications in Specifications D 446.

A1.1.2 Table A1.2 lists certified viscosity reference standards.

TABLE A1.1 Viscometer Types

Viscometer Identification	Kinematic Viscosity Range,[A] mm^2/s
A. Ostwald Types for Transparent Liquids	
Cannon-Fenske routine[B]	0.5 to 20 000
Zeitfuchs	0.6 to 3 000
BS/U-tube[B]	0.9 to 10 000
BS/U/M miniature	0.2 to 100
SIL[B]	0.6 to 10 000
Cannon-Manning semi-micro	0.4 to 20 000
Pinkevitch[B]	0.6 to 17 000
B. Suspended-level Types for Transparent Liquids	
BS/IP/SL[B]	3.5 to 100 000
BS/IP/SL(S)[B]	1.05 to 10 000
BS/IP/MSL	0.6 to 3 000
Ubbelohde[B]	0.3 to 100 000
FitzSimons	0.6 to 1 200
Atlantic[B]	0.75 to 5 000
Cannon-Ubbelohde(A), Cannon Ubbelohde dilution[B](B)	0.5 to 100 000
Cannon-Ubbelohde semi-micro	0.4 to 20 000
C. Reverse-flow Types for Transparent and Opaque Liquids	
Cannon-Fenske opaque	0.4 to 20 000
Zeitfuchs cross-arm	0.6 to 100 000
BS/IP/RF U-tube reverse-flow	0.6 to 300 000
Lantz-Zeitfuchs type reverse-flow	60 to 100 000

[A] Each range quoted requires a series of viscometers. To avoid the necessity of making a kinetic energy correction, these viscometers are designed for a flow time in excess of 200 s except where noted in Specifications D 446.

[B] In each of these series, the minimum flow time for the viscometers with lowest constants exceeds 200 s.

TABLE A1.2 Certified Viscosity Reference Standards

Designation	Approximate Kinematic Viscosity, mm^2/s					
	20°C	25°C	40°C	50°C	80 °C	100°C
S3	4.6	4.0	2.9	...	...	1.2
S6	11	8.9	5.7	...	...	1.8
S20	44	34	18	...	...	3.9
S60	170	120	54	...	...	7.2
S200	640	450	180	...	...	17
S600	2400	1600	520	280	67	32
S2000	8700	5600	1700	...	...	75
S8000	37 000	23 000	6700	...	...	...
S30 000	...	81 000	23 000	11 000	...	...

A2. KINEMATIC VISCOSITY TEST THERMOMETERS

A2.1 Short-Range Specialized Thermometer

A2.1.1 Use a short-range specialized thermometer conforming to the generic specification given in Table A2.1 and Table A2.2 and to one of the designs shown in Fig. A2.1.

A2.1.2 The difference in the designs rests mainly in the position of the ice point scale. In Design A, the ice point is within the scale range, in Design B, the ice point is below the scale range, and in Design C, the ice point is above the scale range.

A2.2 Calibration

A2.2.1 Use liquid-in-glass thermometers with an accuracy after correction of 0.02°C or better, calibrated by a laboratory meeting the requirements of ISO 9000 or ISO 17025, and carrying certificates confirming that the calibration is traceable to a national standard. As an alternative, use thermometric devices such as platinum resistance thermometers, of equal or better accuracy, with the same certification requirements.

A2.2.2 The scale correction of liquid-in-glass thermometers can change during storage and use, and therefore regular re-calibration is required. This is most conveniently achieved in a working laboratory by means of a re-calibration of the ice point, and all of the main scale corrections altered for the change seen in the ice point.

TABLE A2.1 General Specification for Thermometers

NOTE—Table A2.2 gives a range of ASTM, IP, and ASTM/IP thermometers that comply with the specification in Table A2.1, together with their designated test temperatures. See Specification E 1 and Test Method E 77.

Immersion		Total
Scale marks:		
Subdivisions	°C	0.05
Long lines at each	°C	0.1 and 0.5
Numbers at each	°C	1
Maximum line width	mm	0.10
Scale error at test temperature, max	°C	0.1
Expansion chamber:		
Permit heating to	°C	105 up to 90, 120 between 90 and 95 130 between 95 and 105, 170 above 105
Total length	mm	300 to 310
Stem outside diameter	mm	6.0 to 8.0
Bulb length	mm	45 to 55
Bulb outside diameter	mm	no greater than stem
Length of scale range	mm	40 to 90

TABLE A2.2 Complying Thermometers

Thermometer No.	Test Temperature		Thermometer No.	Test Temperature	
	°C	°F		°C	°F
ASTM 132C, IP 102C	150		ASTM 128C, F/IP 33C	0	32
ASTM 110C, F/IP 93C	135	275	ASTM 72C, F/IP 67C	–17.8	0
ASTM 121C/IP 32C	98.9, 100	210, 212	ASTM 127C/IP 99C	–20	–4
			ASTM 126C, F/IP 71C	–26.1	–20
ASTM 129C, F/IP 36C	93.3	200	ASTM 73C, F/IP 68C	–40	–40
ASTM 48C, F/IP 90C	82.2	180	ASTM 74C, F/IP 69C	–53.9	–65
IP 100C	80				
ASTM 47C, F/IP 35C	60	140			
ASTM 29C, F/IP 34C	54.4	130			
ASTM 46C F/IP 66C	50	122			
ASTM 120C/IP 92C	40				
ASTM 28C, F/IP 31C	37.8	100			
ASTM 118C, F	30	86			
ASTM 45C, F/IP 30C	25	77			
ASTM 44C, F/IP 29C	20	68			

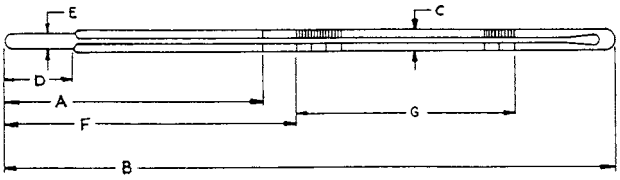
A2.2.2.1 The interval for ice-point recalibration shall be no longer than six months (see NIST GMP 11). For new thermometers, monthly checking for the first six months is recommended. A change of one or more scale divisions in the ice point means that the thermometer may have been overheated or damaged, and it may be out of calibration. Such thermometers shall be removed from service until inspected, or recalibrated, or both. A complete recalibration of the thermometer, while permitted, is not necessary in order to meet the accuracy ascribed to this design thermometer (see NIST Special Publication 819). Any change in ice-point correction shall be added to the other corrections of the original Report of Calibration.

A2.2.2.2 Other thermometric devices, if used, will also require periodic recalibration. Keep records of all recalibration.

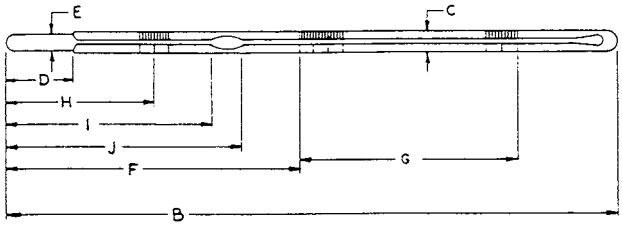
A2.2.3 *Procedure for Ice-point Recalibration of Liquid-in-glass Thermometers*.

A2.2.3.1 Unless otherwise listed on the certificate of calibration, the recalibration of calibrated kinematic viscosity thermometers requires that the ice-point reading shall be taken within 60 min after being at test temperature for not less than 3 min.

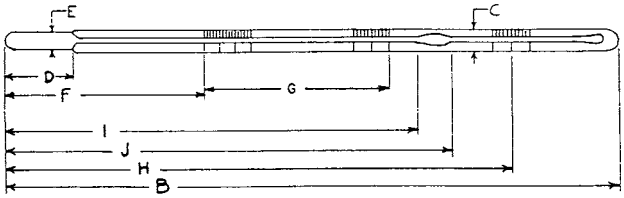
A2.2.3.2 Select clear pieces of ice, preferably made from distilled or pure water. Discard any cloudy or unsound portions. Rinse the ice with distilled water and shave or crush into small pieces, avoiding direct contact with the hands or any chemically unclean objects. Fill the Dewar vessel with the



(a)



(b)



(c)

FIG. A2.1 Thermometer Designs

crushed ice and add sufficient water to form a slush, but not enough to float the ice. As the ice melts, drain off some of the water and add more crushed ice. Insert the thermometer, and pack the ice gently about the stem, to a depth approximately one scale division below the 0°C graduation.

A2.2.3.3 After at least 3 min have elapsed, tap the thermometer gently and repeatedly at right angles to its axis while making observations. Successive readings taken at least 1 min apart shall agree within 0.005°C.

A2.2.3.4 Record the ice-point readings and determine the thermometer correction at this temperature from the mean reading. If the correction is found to be higher or lower than that corresponding to a previous calibration, change the correction at all other temperatures by the same value.

A2.2.3.5 During the procedure, apply the following conditions:

(*1*) The thermometer shall be supported vertically.

(*2*) View the thermometer with an optical aid that gives a magnification of approximately five and also eliminates parallax.

(*3*) Express the ice-point reading to the nearest 0.005°C.

A2.2.4 When in use, immerse the thermometric device to the same depth as when it was fully calibrated. For example, if a liquid-in-glass thermometer was calibrated at the normal total immersion condition, it shall be immersed to the top of the mercury column with the remainder of the stem and the expansion volume at the uppermost end exposed to room temperature and pressure. In practice, this means that the top of the mercury column shall be within a length equivalent to four scale divisions of the surface of the medium whose temperature is being measured.

A2.2.4.1 If this condition cannot be met, then an extra correction may be necessary.

A3. TIMER ACCURACY

A3.1 Regularly check timers for accuracy and maintain records of such checks.

A3.1.1 Time signals as broadcast by the National Institute of Standards and Technology are a convenient and primary standard reference for calibrating timing devices. The following can be used to an accuracy of 0.1 s:

WWV	Fort Collins, CO	2.5, 5, 10, 15, 20 MHz
WWVH	Kauai, HI	2.5, 5, 10, 15, MHz
CHU	Ottawa, Canada	3.33, 7.335, 14.67 MHz

A3.1.2 Radio broadcast of voice and audio on a telephone line at phone 303-499-7111. Additional time services are available from the National Institute of Standards and Technology.

A4. CALCULATION OF ACCEPTABLE TOLERANCE ZONE (BAND) TO DETERMINE CONFORMANCE WITH A CERTIFIED REFERENCE MATERIAL

A4.1 Determine the standard deviation for site uncertainty, σ_{site}, from a laboratory quality control program.

A4.1.1 If the standard deviation for site uncertainty, σ_{site}, is not known, use the value 0.19%.

A4.2 Determine the combined extended uncertainty (CEU) of the accepted reference value (ARV) of the certified reference material (CRM) from the supplier's label or included documentation.

A4.3 Calculate the standard error of the accepted reference value (SEARV) by dividing the CEU by the coverage factor, *k*, listed on the supplier's label or included documentation.

A4.3.1 If the coverage factor, *k*, is not known, use the value 2.

A4.4 Construct the acceptable tolerance zone:

$$TZ = \pm 1.44 \sqrt{\sigma_{site}^2 + SE_{ARV}^2}$$

SUMMARY OF CHANGES

Subcommittee D02.07 has identified the location of selected changes to this standard since the last issue (D 445–04$^{\epsilon 2}$) that may impact the use of this standard.

(*1*) Revised 3.1.1.
(*2*) Revised 6.1.2 and added Note 3.
(*3*) Revised 10.2.
(*4*) Added Note 2.

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STANDARD OPERATING PROCEDURE

SEI-10.15.9

THE DETERMINATION OF VOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY / MASS SPECTROMETRY (GC/MS) (SW846 USEPA METHOD 8260C)

SOP Number: SEI-10.15.9 Date Issued: 08/19/04
Revision Number: 9 Date of Revision: 11/7/12

1.0 OBJECTIVE/

This method, based on SW846 USEPA Method 8260C for the analyses of volatile organic compounds, is designed to measure the concentration of specified volatile organic compounds (VOCs) in water, soil and air samples using solid phase micro-extraction (SPME) and a Gas Chromatograph equipped with a capillary column and mass spectrometer. Specific VOCs of interest are outlined in Table 1.

2.0 SUMMARY OF METHOD

This method provides gas chromatographic conditions for the detection of certain aromatic and chlorinated VOCs. Soil and water samples are prepared in 20 mL extraction vials. The SPME fiber is then exposed to the headspace above the sample for a set number of minutes while the sample is agitated via either stirring or orbital shaking. Air samples are collected in Tedlar bags and the SPME fiber is exposed directly to the sample through the syringe septum. The exposure time must be consistent for all standards and samples (typically six or eight minutes). The absorbed analytes are thermally desorbed from the SPME fiber in the injection port of a Gas Chromatograph and transferred to a narrow bore capillary column. A temperature program is used to enhance separation as the analytes are carried through the column to the ion source of the mass spectrometer. The ionized molecules are focused and separated according to their mass/charge (m/z) ratio by a quadrupole analyzer. The signal is amplified by an electron multiplier and interpreted by the mass spectrometer data system to produce a total ion or selected ion chromatogram and mass spectra for every point on the chromatogram. The instrument's mass spectrometer can be set in scan mode or selective ion monitoring mode (SIM). SIM mode is typically used to achieve lower reporting limits.

This method is suitable for analysis of air, water, soils, and wastes extractable in methanol.

Soil and waste samples are collected in approximately 10-12 gram cores directly into methanol (10 mL) in 40 mL VOA vials. Subsequent to sample collection, the methanol/solid mixture is agitated to break up the solids. Following this extraction step, an aliquot of the methanol is diluted into analyte-free water and is further extracted via the headspace SPME technique. Water samples are collected in 40 mL VOA vials with no headspace. All soil and water samples are analyzed as waters on the GC/MS.

This method is based on USEPA 8260C, *Volatile Organic Compounds by Gas Chromatography Mass Spectrometry (GC/MS)* and ASTM Method D6520, *Solid Phase Micro-extraction of water and its headspace for analysis of Volatile and Semi-volatile Organic Compounds*.

3.0 DEFINITIONS

Accuracy: the degree of agreement between a measurement and true or expected value, or between the average of a number of measurements and the true or expected value.

Batch: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of a similar matrix, meeting the above-mentioned criteria.

Corrective Action: action taken to eliminate the causes of an existing non-conformance, defect or other undesirable situation in order to prevent recurrence.

Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.

Instrument Blank: a blank matrix that is the same as the processed sample matrix (i.e. extract, digestate, condensate) and introduced onto the instrument for analysis. Analyzed typically as a fiber blank.

Laboratory Duplicate: Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently. (NELAC)

Limit of Detection (LOD): An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte- and matrix-specific and may be laboratory-dependent. (DOD QSMV4.2)

Limit of Detection (Clarification): The smallest amount or concentration of a substance that must be present in a sample in order to be detected at a high level of confidence (99%). At the LOD, the false negative rate (Type II error) is 1%. (DOD QSMV4.2)

Limits of Quantitation (LOQ): The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. (DOD QSMV4.2)

Limit of Quantitation (Clarification): The lowest concentration that produces a quantitative result within specified limits of precision and bias. For DoD projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

Matrix: the substrate of a test sample.

Matrix Duplicate (MD): duplicate aliquot of a sample processed and analyzed independently; under the same laboratory conditions; also referred to as Sample Duplicate; Laboratory Duplicate.

Matrix Spike (MS): field sample to which a known amount of target analyte(s) is added.

Matrix Spike Duplicate (MSD): a replicate matrix spike.

Method Blank: a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

Precision: an estimate of variability. It is an estimate of agreement among individual measurements of the same physical or chemical property, under prescribed similar conditions.

Method Detection Limit: One way to establish a Limit of Detection, defined as the minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. (DOD QSMV4.2)

Method Detection Limit (MDL) (Clarification): The MDL is one way to establish a Detection Limit, not a Limit of Detection. (DOD QSMV4.2)

Nonconformance: An indication or judgment that a product or service has not met the requirement of the relevant specifications, contract, or regulation; also the state of failing to meet the requirements. (DOD QSMV4.2)

Precision: The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms. (NELAC)

Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical and/or biological integrity of the sample. (NELAC)

Proficiency Testing: A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source. (NELAC) [2.1]

Quality Assurance (QA): an integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence.

Quality Control (QC): the overall system of technical activities, the purpose of which is to measure and control the quality of a product or service.

Quality Control Sample: a control sample, generated at the laboratory or in the field, or obtained from an independent source, used to monitor a specific element in the sampling and/or testing process.

Quantitation Limit (QL): the minimum amount of a substance that can be quantitatively measured with a specified degree of confidence and within the accuracy and precision guidelines of a specific measurement system. The QL can be based on the MDL, and is generally calculated as 3-5 times the MDL, however, there are analytical techniques and methods where this relationship is not applicable. Also referred to as Practical Quantitation Level (PQL), Estimated Quantitation Level (EQL), Limit of Quantitation (LOQ).

Quantitation Range: The range of values in a calibration curve between the LOQ and the highest successfully analyzed initial calibration standard. The quantitation range lies within the calibration range. (DOD QSMV4.2)

Raw Data: Any original factual information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments. If exact copies of raw data have been prepared (e.g., tapes which have been transcribed verbatim, data and verified accurate by signature), the exact copy or exact transcript may be submitted. (EPA-QAD)

Reporting Limit (RL): The level to which data is reported for a specific test method and/or sample. The RL is generally related to the QL. The RL must be minimally at or above the MDL.

Requirement: Denotes a mandatory specification; often designated by the term "shall". (NELAC)

Retention Time: The time between sample injection and the appearance of a solute peak at the detector. (Skoog, West, and Holler. Fundamentals of Analytical Chemistry. 1992)

Sample: Portion of material collected for analysis, identified by a single, unique alphanumeric code. A sample may consist of portions in multiple containers, if a single sample is submitted for multiple or repetitive analysis. (DOD QSMV4.2)

Selectivity: (Analytical chemistry) The capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances. (EPA-QAD)

Selective Ion Monitoring: A configuration of the mass spectrometer in which the intensities of several specific ion beams are recorded rather than the entire mass spectrum.

Sensitivity: The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (NELAC)

Shall: Denotes a requirement that is mandatory whenever the criterion for conformance with the specification requires that there be no deviation. This does not prohibit the use of alternative approaches or methods for implementing the specification so long as the requirement is fulfilled. (ANSI)

Should: Denotes a guideline or recommendation whenever noncompliance with the specification is permissible. (ANSI)

Signal to Noise Ratio: The signal carries information about the analyte, while noise is made up of extraneous information that is unwanted because it degrades the accuracy and precision of an analysis and also places a lower limit on the amount of analyte that can be detected. In most measurements, the average strength of the noise is constant and independent of the magnitude of the signal. Thus, the effect of noise on the relative error of a measurement becomes greater and greater as the quantity being measured (producing the signal) decreases in magnitude. (Skoog, Holler, and Nieman. Principles of Instrumental Analysis. 1998)

Spike: a known amount of an analyte added to a blank, sample or sub-sample.

Standard: (Document) The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of NELAC and meets the approval requirements of NELAC procedures and policies. (ASQC)

Standard: (Chemical) Standard samples are comprised of a known amount of standard reference material in the matrix undergoing analysis. A standard reference material is a certified reference material produced by the US National Institute of Standards and Technology (NIST) and characterized for absolute content, independent of analytical test method.

Standard Method: A test method issued by an organization generally recognized as competent to do so.

Standard Operating Procedure (SOP): a written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks.

SPME – Solid Phase Micro Extraction

Surrogate: A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes. (QAMS)

Target Analytes: Analytes specifically named by a client (also called project-specific analytes). (DOD QSMV4.2)

Technical Director: Individual(s) who has overall responsibility for the technical operation of the environmental testing laboratory. (NELAC)

Tedlar Bag – Inflatable sample container, used to trap air samples for analysis, equipped with a Teflon®-lined syringe septum that allows a SPME fiber to come in contact with the air sample.

Traceability: The property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons. (VIM - 6.12)

Tuning: A check and/or adjustment of instrument performance for mass spectrometry as required by the method.). (DOD QSMV4.2)

VOC – Volatile Organic Compounds

QA/QC – Quality Assurance/Quality Control

4.0 INTERFERENCES

When analyzing for VOCs, samples can be contaminated by diffusion of volatile organics (particularly chlorofluorocarbons and methylene chloride) through the sample container septum during shipment and storage. A trip blank can be used as a check on this type of interference however, a trip blank is not usually required if the analyses are being conducted onsite. A trip blank is prepared from analyte-free reagent water and carried through sampling and subsequent storage and handling.

Contamination by carryover can occur whenever high-concentration and low-concentration samples are analyzed in sequence. To reduce the potential for carryover, the sample syringe or purging device must be

rinsed out between samples with an appropriate solvent. Whenever an unusually concentrated sample is encountered, it should be followed by injection of a method blank to check for cross contamination.

Purging vessels and syringes should be adequately cleaned and flushed prior to use. All glassware must be kept scrupulously cleaned. Clean all glassware as soon as possible after use by rinsing with the last solvent used or analyte-free water. Store clean dry glassware in a clean environment.

5.0 SAFETY ISSUES

1. Laboratory personnel should take extra care when working with standards. When working with neat standards, make certain that there is adequate ventilation and that Nitrile or Latex gloves are worn and if necessary, safety glasses.
2. If necessary and appropriate, a site-specific health and safety plan shall be created for each study site and followed.
3. Field visits may involve accessing remote areas. Health and safety concerns regarding these field visits are minimal, however, hazards such as slip, trip and falls, poisonous plant and dangerous animals, as well as getting vehicles stuck in remote areas, do present considerable health and safety issues. To help ensure field staff's health and safety in remote areas, all field staff are to have daily communication with the project manager or another appropriate SEI employee.
4. All chemicals are required to be received with a Material Safety Data Sheets (MSDS). MSDSs shall be made available to all personnel involved in the sampling and testing.

6.0 EQUIPMENT AND SUPPLIES

General maintenance shall be conducted proactively to minimize the down time associated with equipment failure and to maintain the highest degree of data quality. This shall be accomplished through scheduled and symptomatic maintenance. Examples of symptomatic maintenance, in which the chromatography is monitored, include cleaning of inlet, replacement of septa and liner and mass spectrometer source cleaning. Examples of scheduled maintenance include replacement of diffusion and rough pump oils. All maintenance conducted on equipment shall be documented in the instrument maintenance logs. The primary pieces of instrumentation for the laboratory are listed below.

1. Gas Chromatograph: Analytical system complete with gas chromatograph and all required accessories, column supplies, gases, syringes and data system to determine peak areas and perform integrations. Hewlett Packard 5890 Series II.
2. Mass Spectrometer: Hewlett Packard 5971 MSD.
3. Typical Analytical Column: RTX-VMS 20m x 0.18mm ID, 1.0 um film or equivalent
4. SPME Autosampler: CTC Combi PAL

5. Array of micro-syringes: range 5-1000 uL.
6. Balance: Top loading, capable of weighing accurately to 0.01 grams (soil samples only).
7. Sample introduction and preparation apparatus: SPME Fiber type 100 µm polydimethylsiloxane (PDMS) and/or 85 µm Carboxen/PDMS fiber, magnetic hot plate and magnetic stirrers, tube clamp and stand.
8. VOA vials: 40 mL collection containers with Teflon®-lined septum and either 24mL or 20mL extraction vials

7.0 REAGENTS AND STANDARDS

Reagents:

1. Analyte-free reagent water demonstrated to be free of compounds of interest (spring water, carbon filtered and/or deionized).
2. Purge and Trap Grade or equivalent grade methanol (demonstrated to be free of analytes and stored away from other solvents).

Stock standards:

Stock standards may either be prepared from pure standard materials or purchased as certified solutions. Secondary dilution standards are prepared using stock standard solutions, which contain the compounds of interest, either as single compounds or mixed together. Typically, for this method the stock standards are obtained from an approved vendor and mixed together. A certificate of analysis is retained by the laboratory and maintained on file or on file with the vendor. The aqueous standards are typically prepared at concentrations as listed in Section 9.0 and are prepared in accordance with Table 3 which is an Example of the Standard Preparation Calculation Sheet. Gas standards are purchased in mixtures of site-specific target analytes.

Calibration standards are prepared at a minimum of five to seven different concentrations levels for VOC analytes.

Stone's SOP No. SEI-4.7.n *Labeling, Preparation and Storage of Reagents, Solutions and Standards* provides procedures on properly labeling, preparing and storing of reagents and standards used in the mobile laboratory for analytical methods. This SOP should be reviewed by the analyst before labeling, preparing and storing standards within the mobile laboratory.

8.0 SAMPLE COLLECTION AND HANDLING

8.1 Sample Collection

1. Aqueous samples are collected in 40 mL Teflon®-lined septum VOA vials filled to ensure that no headspace is present. Upon receipt, and if multiple vials are presented for a given sample, the vials will be uniquely numerically labeled (with the numbers 1, 2, 3, etc.) and

at the time of analysis this unique identifier will be recorded in the instrument's run log. If storage of these samples is expected to exceed seven days, aqueous samples should be preserved with hydrochloric acid (HCl) to a pH of < 2.

2. Soil and waste samples are collected in approximately 10-12 gram cores, and placed in 40 mL vials with 10 mL of methanol. Upon receipt, and if multiple vials are presented for a given sample, the vials will be uniquely numerically labeled (with the numbers 1, 2, 3, etc.) and at the time of analysis this unique identifier will be recorded in the instrument's run log. If required, a separate aliquot of soil from the sample location will be collected in a 40 mL vial (with no methanol preservative) for percent moisture determination.

3. Air samples are collected in Tedlar bags. Upon receipt, and if multiple bags are presented for a given sample, the bags will be uniquely numerically labeled (with the numbers 1, 2, 3, etc.) and at the time of analysis this unique identifier will be recorded in the instrument's run log. The analytes are concentrated on the SPME fiber by exposing the fiber directly to sample through the Teflon®-lined septum. Air samples must be analyzed within 3 days of collection.

8.2 Handling and Holding Times

1. Unpreserved water samples must be analyzed within 7 days of collection. Preserved (preserved with HCl to a pH of < 2) water samples must be analyzed within 14 days of collection. Soil samples collected in methanol must be analyzed within 14 days from collection. All samples shall be stored at < 6°C. Samples are allowed to come to room temperature just prior to analysis. All samples for this program are typically analyzed within one or two days from collection, therefore, holding times should not be a concern.

2. If samples were preserved and analyses are performed after 7 days from collection, pH determination (water samples only) should be performed. Once the sample aliquots have been taken from the VOA vial for analysis, the pH of the water sample must be determined. The purpose of the pH determination is to ensure that all VOA samples were acidified in the field. Test the pH by placing one or two drops of sample on the pH paper (do not add pH paper to the vial). Record the pH of each sample, and report these data on the runlog and in the SDG Narrative. No pH adjustment is to be performed.

3. If necessary, a separate soil sample aliquot will be collected in a 40 mL vial or equivalent for percent moisture content so that results can be adjusted for %moisture and reported as dry weight rather than as received. Percent moisture is determined as follows: Percent Moisture Determination - Immediately after weighing the sample for analysis, weigh 5-10 g of the soil/sediment into a tared crucible. Determine the percent moisture by drying overnight at 105 ˚C. If available, allow to cool in a desiccator before weighing. Concentrations of individual analytes will be reported relative to the dry weight of soil/sediment.

$$\text{Percent Moisture} = \frac{\text{Mass of wet sample (g)} - \text{Mass of dry sample (g)}}{\text{Mass of wet sample (g)}}$$

4. If samples are suspected to be of high concentration and are not to be immediately analyzed, they will be isolated in a separate cooler unit in the lab. If a separate cooler is not readily available in the lab, one will be purchased.

9.0 PROCEDURES

9.1 Typical GC Program

Oven Temp: 50°C (4.00 min) to 160°C at 15°C/min to 200°C at 40°C/min, hold for 1 min.

Flow Rate: Helium, 1.0 mL/min, 8 PSI, set at 45°C

Detector: MSD, SIM mode, ion masses range between 45-190 amu

Injector: 240°C, splitless (close 1.5 min)

Liner: 0.75 mm ID splitless/split injection port

9.2 GC Column

Column: RTX-VMS 20m x 0.18mm ID, 1.0 um film, equivalent

9.3 Material Preparation:

SPME: Fiber Type: 100 µm polydimethylsiloxane (PDMS).

SPME Fiber Type for low level waters or specialized project only: 85 µm Carboxen/PDMS

SPME Extraction: The SPME-absorbent polymer fiber is exposed to the headspace above a sample. The headspace is created by removing approximately 2 mLs from the 20 mL preparation vial. Salt and stir bar (if stirring is used) are added to the mixture and the sample is agitated for 6 or 8 minutes. The fiber is then retracted into the SPME holder and transferred to the GC injection port where the analytes are thermally desorbed and transferred to the head of the GC column. If used, each magnetic stirrer is rinsed three times with analyte free water prior to use. For soil samples; an aliquot of the methanol extract (typically 0.5mL but not more than 1 mL) is prepared in the 20 mL water preparation vial, and prepared and analyzed in the same manner as a water sample above. For air samples, the SPME fiber is exposed directly to the same through the syringe septum located on the Tedlar bag. Preparation and exposure time of the fiber to the site samples is the same for all QC samples and calibration standards.

9.4 Operating Procedure

9.4.1 Tuning with 4-Bromofluorobenzene

The following ion abundances must be met before analysis of calibration standards, environmental samples and at the beginning of a 12-hour analytical sequence by analyzing BFB in a check standard, method blank or as a single injection:

BFB Key Ions and Ion Abundance Criteria	
Mass	Ion Abundance Criteria
50	15.0-40.0 percent of mass 95
75	30.0-60.0 percent of mass 95
95	base peak, 100 percent relative abundance
96	5.0-9.0 percent of mass 95
173	less than 2.0 percent of mass 174
174	>50.0 percent of mass 95
175	5.0-9.0 percent of mass 174
176	95.0-101.0 percent of mass 174
177	5.0-9.0 percent of mass 176

9.4.2 Calibration Criteria

9.4.2.1 Initial Calibration (ICAL):

1. Five to seven-point calibrations for VOC compounds using a SIM configuration is as follows: 2, 5, 10, 20, 50, 100, 200 ppb. (Scan calibrations are determined as needed by the client's program) The low standard must be set at the quantitation limit (QL) or LOQ. A typical calibration range for air ($ppb^{v}/_{v}$) will be similar to that of water and will be chosen on a site specific basis.
2. Calibration range must encompass the expected sample concentrations; therefore, if site samples are expected to be higher in concentration than the calibration range, adjustments to the calibration range and samples will be documented and made accordingly.
3. The linearity of the calibration curves must be assessed and are used for all quantitation. Volatile organic compounds in soil and water use an internal standard procedure (internal standards; fluorobenzene and chlorobenzene-d5). Average response factors are used for quantitation and the average relative standard deviation (%RSD) of each compound must be ≤ 20%. Linear least squares regression must be r > 0.995
4. Non-linear regression coefficient of determination (COD) must be r2 > 0.99 (6 points shall be used for second order, 7 points shall be used for third order). Depending upon

the field on-site program, 25% RSD may be acceptable based on the on-site data quality objectives.

For DoD Projects (DOD QSM4.2, Table F-4), one of the following must be true for each analyte in the calibration:

- % RSD for each analyte must be ≤15% (method requirement ≤ 20%).
- Linear least squares regression must be r ≥ 0.995
- Non-linear regression coefficient of determination (COD) must be $r^2 \geq 0.99$ (6 points shall be used for second order, 7 points shall be used for third order).

Note: The average response factor requirements are only for 8260B and were not included in 8260C, therefore, for DOD projects the laboratory will follow the updated method. (The average response factor requirement will be ≥0.30 for chlorobenzene and 1,1,2,2-tetrachlorethane, and ≥0.1 for chloromethane, bromoform, and 1,1-dichloroethane (SPCCs). % RSD must be ≤30% for CCCs if analyzing for 8260B only).

5. VOCs in air use an external calibration procedure. Linear regression is used for quantitation and the correlation coefficient of each compound must be greater than 0.99

9.4.2.2 Continuing Calibration (CCV):

1. A mid-point continuing calibration verification standard for VOC compounds (20 ppb or 50ppb) must be analyzed every 12 hours, prior to sample analysis in order to verify the initial calibration. A percent difference between the expected concentration and actual concentration is calculated. The percent difference (%D) values for each compound must be less than or equal to 20%.

$$\%D = \frac{ExpectedConcentration - ActualConcentration}{ExpectedConcentration}$$

The CCV may also be used to demonstrate that the BFB criteria shown in 10.4.1 have been met when operating in scan mode.

9.4.2.3 Initial Calibration Verification Standard (ICV):

A QC sample from a second (independent) source will be analyzed directly after the ICAL. The ICV sample will contain all the VOC compounds of interest at the mid-point (20-100 ppb or 5 ppb for low level waters). Recovery Limits for the ICV will be 70-130%, if compounds are determined to be poor performers then the recoveries shall be 60-140% for the ICV.

For DoD projects, all project analytes must be within tolerances liststed in Table 2.

$$\%\text{Recovery} = \frac{\text{Actual Concentration}}{\text{Expected Concentration}}$$

9.4.3 Retention Time Windows and Pattern Recognition

The laboratory shall identify single component target analytes by comparison of its mass spectrum to a reference spectrum. The retention time for each analyte should be within ± 0.04 minutes (DOD QSM4.2, Table F-4 allows +/-0.06 minutes) from the ICAL or daily CCV.

9.4.4 Internal Standard Response

Internal standard areas shall be evaluated based on the value that was observed in the midpoint of calibration curve. The internal standard areas shall be less than ½ or greater than two times the area found in the midpoint of the curve.

9.4.5 Sample Preparation:

Water samples: An aliquot of sample, sufficient to get the highest concentration constituents into the upper half of the linear range, is transferred to a 20 mL extraction vial which contains approximately ¼ tsp. salt. The vial is then spiked with the surrogate (bromofluorobenzene between 4.17 and 41.67 ppb) and internal standard (fluorobenzene between 4.17 and 41.67 ppb). Invert the vial a few (approximately 3) times and remove approximately five mLs and place the vial on the autosampler tray. The autosampler tray is then placed on the CTC autosampler and set for analysis in accordance with the CTC specifications. At that point the CTC autosampler agitates the sample and the SPME fiber is then exposed to the heads+pace (headspace created by removing approximately 5 mL from preparation vial) above the sample for 6 or 8 minutes while the sample is agitated. The 6 or 8 minutes is measured by either the autosampler. Extraction time may be modified depending on the anticipated equilibration response of the target analytes at given concentrations (if adjusted samples and standards would be adjusted).

Soil samples: Preserved in methanol; 10-12 g core of soil is added to 10 mL of methanol containing an appropriate amount of bromofluorobenzene. Subsequent to sample collection, the methanol/soil mixture is agitated to break up the solids. The level of agitation shall be sufficient enough so that the solids are completely disaggregated as to maximize the contact between the methanol and the soil surfaces. This is typically achieved within 10 minutes using an aggressive orbital shaking procedure. Following this extraction step, an aliquot of the methanol, sufficient to get the highest concentration constituents into the upper half of the linear range but not to exceed 2 mLs, is diluted into analyte-free water and this water/methanol mixture is further extracted via the headspace SPME technique in the same manner as a water sample.

Air samples: Exposure of the SPME fiber to the air sample occurs directly through the syringe septum in the Tedlar bag. Exposure time, measured using a bench-top timer, may be modified depending on the anticipated equilibration response of the target analytes at given concentrations. There is no surrogate or internal standard for air sampling.

9.4.6 Analysis of Samples:

Analysis of samples is performed using SPME followed by analysis on a GC equipped with a narrow bore capillary column and a MSD. For this program the fiber is exposed to the headspace above the sample in the CTC autosampler. A small portion of sample is discarded from the 20 ml vial prior to exposure to generate headspace (approximately 5 mLs). The exposure time needs to be consistent (typically 6 to 8 minutes for this method). To facilitate the release of the analytes from their respective media, the sample is agitated throughout the extraction process (fiber exposure). Following extraction, the fiber adsorbed analytes are thermally desorbed within the injection port of the GC and are transferred to the capillary column.

9.4.7 Surrogate (SS) and Internal Standard (ISTD) Compounds for VOC compounds

Compound	**Amount Spiked**
Bromofluorobenzene (SS)	Range: 4.17 to 41.67 ppb
Fluorobenzene (ISTD)	Range: 4.17 to 41.67 ppb
Chlorobenzene-d5 (ISTD)	Range: 4.17 to 41.67 ppb

9.4.8 LCS Preparation

Compounds and spike amounts are on Table 1. For a waters program, compounds and spike amounts are specified according to project specific needs. Waters are spiked at around the mid-point of the calibration.

For the soils program, the soil LCS will be prepared in the same manner as the soil samples, an aliquot (0.5 mL or 1.0 mL) of methanol will be prepared in the SPME preparation vial, spiked with VOCs of interest, and prepared in the same manner as the soil MS/MSD.

9.4.9 Matrix Spike and Matrix Spike Duplicate (MS/MSD) Compounds:

For water samples the MS in SIM configuration will be spiked at 20 ppb; the amount may change to account for the concentration of a particular analyte found in the sample being analyzed as a MS. For soil samples, an appropriate amount of the target analytes are spiked

into the 10 mL of methanol. Matrix Spike Analyses contains the VOC compounds of interest in the same manner as the LCS.

9.4.10 Target Compound List Compounds

A Target Compound List with Quantitation Limits can be found on Table 1.

10.0 CALCULATIONS

Identification of the project specific VOCs in the sample chromatogram is based on retention time and on comparison of the sample mass spectrum with characteristic ions in a reference mass spectrum. Retention times must be within ± 0.04 minutes from the ICAL. If a retention time shift is observed in the CCV or daily ICV, then adjustments to the daily retention time windows will be made accordingly. The relative intensities of the characteristic ions must agree within 30% of these ions in the reference spectrum. Quantification of the VOCs is determined by comparison to the internal standard and average response factor for the target ion of the analyte.

The concentration of each analyte in the sample may be determined by calculating the amount of standard injected, from the target ion peak response, using the calibration curve. The calculations are as noted below.

Concentrations of Unknowns

Method 8260B does not include reporting of tentatively identified compounds (TIC). However, reporting of up to 10 TIC's whose peak heights are ≥ 10% of the nearest internal standard may be requested or ≥ 40% of the nearest internal standard may be requested for low concentration analyses only when the laboratory has analyzed the samples in Scan Mode. Concentrations are calculated using the following formula using an assumed response factor (RF) of 1:

$$C_{(x)} = \frac{C_{(IS)} * A_{(x)}}{A_{(IS)}} * DF$$

Where:

$C_{(x)}$= Concentration of Unknown (µg/L).

$C_{(IS)}$= Concentration of internal standard (µg/L).

DF = Dilution Factor

$A_{(x)}$ = Area of Unknown (from the total ion chromatogram)

$A_{(IS)}$= Area of associated internal standard (from the total ion chromatogram).

Concentrations of Calibrated Compounds (when RRF is used):

$$C_{(x)} = \frac{A_{(x)} * C_{(IS)}}{A_{(IS)} * \overline{RRF}} * DF$$

Where:

$C_{(x)}$ = Concentration of compound (µg/L)

$C_{(IS)}$ = Concentration of associated internal standard (µg/L).

DF = Dilution Factor.

$A_{(IS)}$ = Area of quantitation ion for associated internal standard.

$A_{(x)}$ = Area of quantitation ion for compound.

RRF = Average Relative Response Factor from five-point initial calibration.

Note: for linear and quadratic equations see SW846 Method 8000C.

Data Reporting - Based on the mass spectra and the project specific field program, it is appropriate to report values between the MDL/Limit of detection (LOD) and the QL or LOQ. In this region, an analyte can be qualitatively detected, but not accurately quantified. Any data point reported in this region is flagged with a "J" (estimated) qualifier. Stone reports sample specific QL's. Sample specific QL's are derived by taking into account various sample specific data, which can include the amount of the sample subject to testing, % moisture, dilution factor, interferences and the base QL's for the analysis.

Typical Reporting qualifiers are as follows:

B = Analyte is found in the associated method blank as well as the sample.

D = Compound is identified in an analysis at a secondary dilution factor.

E = Compound quantitation is above the instrument's calibration range for this analysis.

J = Indicates an estimated quantitation value.

Q = One or more quality control criteria failed (e.g., LCS recovery, surrogate spike recovery or CCV).

U = Compound was analyzed for but not detected. Value reported is the method LOQ.

11.0 QUALITY CONTROL AND CORRECTIVE ACTION

11.1 Limit of Detection Studies

Limit of Detection (LOD) studies must be completed initially for each method, matrix and instrument and then verified quarterly for each method, lab and matrix. The initial LOD study is the same as the MDL study. LOD verifications are accomplished by running a spiked standard at a value that is greater than 1 to 4 times the LOD. Since we have a range of LODs, we will likely have to run several LOD verifications.

11.2 Limit of Quantitation

Limit of Detection (LOQ) studies must be completed initially for each method, matrix and instrument and then verified quarterly for each method, instrument and matrix. The initial LOQ study is conducted by running the low point of the calibration curve back through the curve once the curve is built in the data analyses software. A successful study is one where the recovery of each analyte is within the established test method acceptance criteria or client data quality objectives for accuracy. The LOQ results must be within the limits of the DOD's limits for LCS recoveries, as stated in Table 2.

11.3 Measurement of Uncertainty

Measurement of Uncertainty studies must be completed initially for each method, matrix and instrument and then verified quarterly for each method, instrument and matrix. These are accomplished by looking at the deviation of recovery for each analyte in an LCS. For example, a recovery of 97% would have a 3% uncertainty associated with it.

11.4 Instrument Blank (SPME Fiber Blank)

To be performed once daily at the start of the analytical day. All target analyte areas detected in the instrument blank must be less than one-half the area of the analyte at the PQL value prior to continuing.

11.5 MS Tuning - 4-Bromofluorobenzene

To be performed every 12 hours, at the start of the analytical day and before beginning acquisition of a calibration curve. Criteria can be found in section 9.4.1.

11.6 Initial Calibration

Prior to the analysis of any samples or QC samples an initial calibration curve for the quantification of analytes of interest is established using criteria as noted in Section 9.0. Minimum of 5 calibration standards with the lowest standard concentration at or below the RL. %RSD for all analytes ≤20%RSD. Least squares regression r ≥ 0.995; non-linear regression– coefficient of determination (COD) r2 ≥ 0.99 (6 points shall be used for second order, 7 points shall be used for third order). If this criteria is not met, then correct problem then repeat ICAL.

Calibration range must encompass the expected sample concentrations; therefore, if site samples are expected to be higher in concentration than the calibration range, adjustments to the calibration range and samples will be documented and made accordingly.

11.7 Continuing Calibration Verification

To be performed every 12 hours, at the start of the analytical day. Percent differences or percent drift must be less than or equal to ≤ 20%.

11.8 Method Blank

One per analytical batch (12 hours or 20 samples) directly after the calibration standard and after highly contaminated samples to demonstrate that there is no carry over into subsequent samples. All target analyte concentrations detected in the method blank must be less than one-half the PQL value prior to continuing.

For DoD projects, the method blank will be considered to be contaminated if:

- The concentration of any target analyte in the blank exceeds ½ the reporting limit **and** is greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater);
- The concentration of any common laboratory contaminant in the blank exceeds the reporting limit **and** is greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater); or
- The blank result otherwise affects the samples results as per the test method requirements of the project-specific objectives.

If the method blank is contaminated as described above, then the laboratory shall reprocess affected samples in a subsequent preparation batch, except when sample results are below the limit of detection (LOD). If insufficient sample volume remains for reprocessing, the results shall be reported with appropriate data qualifiers.

11.9 Surrogate Standards

Surrogate recoveries should be 70-130 % in water samples and 60-140 % in soil samples. Samples with surrogate recoveries falling outside of quality control limits should be reanalyzed once. If the reanalysis of the sample is also outside the quality control limits, then it will be determined that the problem may be associated with the sample matrix and noted as such.

For DoD projects, the acceptance limits for 4-Bromofluorobenzene as a surrogate are 75-120%.

11.10 Internal Standards

Internal standard areas shall be evaluated based on the value that was observed in the midpoint of calibration curve. If the internal standard area is less than ½ or greater than two times the area found in the midpoint of the curve, the affected sample(s) should be rerun to confirm a possible matrix affect.

11.11 Initial Calibration Verification Standard (ICV)

A QC sample from a second source independent of the initial calibration will be analyzed directly after the ICAL or CCV. The ICV sample will contain all the VOC compounds of interest at mid point of the calibration curve. Recovery Limits for the ICV will be 70-130% and 40-160% for poor performers. For DoD projects, the ICV Recovery Limits will be 80-120%. This sample may also be used as the laboratory control sample (LCS).

11.12 Laboratory Control Sample (LCS)

For a waters program, compounds and spike amounts are specified according to project specific needs. Waters will be spiked at around the mid-point of the calibration. For the soils program, the soil LCS will be prepared in the same manner as the soil samples, an aliquot (0.5 mL) of methanol will be prepared in the SPME preparation vial, spiked with VOCs of interest, and prepared in the same manner as the soil MS/MSD. Recovery Limits for the LCS are as specified in Table 2 (DOD LCS Limits) and for compounds not in Table 2 the limits will be 60-140%. These shall be analyzed on the frequency of one per analytical batch. For DoD projects, all target analytes must be spiked in the LCS. If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.

11.13 Matrix Spike and Matrix Spike Duplicate (MS/MSD) Analysis

An MS/MSD analysis is required every set of twenty samples or as specified by the client. A sample may be chosen by the client to perform a MS/MSD analysis.

Recovery Limits for the MS/MSD are as specified in Table 2 (DOD LCS Limits) and for compounds not in Table 2 the limits will be 60-140%. For DoD projects, all target analyes must be spiked in the MS/MSD samples.

$$\%\text{Recovery} = \frac{\text{Sample Spiked Concentration} - \text{Un-Spiked Sample Concentration}}{\text{Spiked Concentration}}$$

11.14 CORRECTIVE ACTION

Quality Control Check	*Minimum Frequency*	*Acceptance Criteria*	*Corrective Action*
Initial Calibration (ICAL)	ICAL prior to sample analysis.	Minimum of five to six points for VOCs. Minimum RRF and Maximum %RSD on Table 1 or a correlation factor (R or R^2) of 0.99 for other curve types. Air calibration's linear regression should have correlation factor of 0.99.	Verify solution integrity and check instrument performance. Perform necessary maintenance and recalibrate instrument. Reanalyze all affected samples.
Initial Calibration Verification (ICV) and/or Laboratory Control Sample (LCS)	One ICV per calibration (following acceptable ICAL). One LCS per analytical batch.	QC limits are 70 to 130% for target compounds in water and soil analyses. This same standard may be used as the daily LCS in which the QC limits are set to 70-130% for target compounds and 60-140% for poor performers. All analytes for air analyses should be within 60-140%. For DoD projects, the ICV Recovery Limits will be 80-120%. This sample may also be used as the laboratory control sample (LCS) Note: Limits may be adjusted based on the program.	Reanalyze once; if still out verify solution integrity or ICAL solution integrity, and instrument performance. Re-prepare and reanalyze all associated samples.
Retention Time (RT) Window Study	Every new column installation/per ICAL	All target compounds and surrogates in all standards must fall within the established window of ± 0.04 minutes from ICAL	Perform system maintenance. Reanalyze affected samples.
Continuing Calibration Verification (CCV)	Daily before sample analysis and every 12 hours of analysis time.	See Section 9.0. ± 20%D for VOCs in water and soils programs. ± 30%D for VOCs in air program.	Verify solution integrity and instrument performance. Reanalyze standard once, if still out, recalibrate and reanalyze affected samples. Based on project specific objectives, marginal exceedances may be allowed. If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since last acceptable CCV.

Quality Control Check	*Minimum Frequency*	*Acceptance Criteria*	*Corrective Action*
Method Blanks	One per preparation batch	Target Compounds < one-half reporting limit. See section 11.4 above for DOD criteria.	Investigate source of contamination. Re-prepare and reanalyze all associated samples. If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.
Matrix Spike (MS) and Matrix Spike Duplicate (MSD)	One set per 20 samples of a similar matrix or as specified by the Client.	Recovery Limits for the MS/MSD are as specified in Table 2 (DOD LCS Limits) and for compounds not in Table 2 the limits will be 60-140%. RPD not to exceed 30% for waters RPD not to exceed 50% for soil samples.	Reanalyze only if recoveries indicate analytical error. If LCS or ICV are acceptable, narrate as possible matrix effect. For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and put in the case narrative.
Tuning – 4-Bromofluorobenzene	every 12 hours or at the start of a new analytical day, and before acquisition of a new calibration curve	See section 9.4.1. Not applicable for air program.	Reanalyze once, if still out, MS should be retuned and adjusted to meet all criteria before continuing
Surrogate Recovery	Each sample	70-130% in water and 60-140% for soil samples For DOD projects 75-125% for water and soil samples	Rerun the sample once, if still out verify the solution integrity and instrument performance. Apply Q-flag or other flag to all associated analytes if acceptance criteria are not met.
Internal Standard Verification	Every field sample, standard, and QC sample.	Retention time ± 30 seconds from retention time of the midpoint standard in the ICAL; EICP area within -50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples analyzed while system was malfunctioning is mandatory. If corrective action fails in field samples, apply Q-flag to analytes associated with the non-compliant IS. Flagging criteria are not appropriate for failed standards.

11.15 Data Review

The analyst is responsible for primary data review of data generated from the sample analysis. Analyses will be documented in the instrument run log. Maintenance is documented in the Instrument Maintenance Logbook. Instrument calibrations and recoveries of all QC samples must be within specified control limits. If instrument calibration or the recoveries of any QC sample exceed specified tolerances, then the affected sample results are evaluated and generally the samples are

submitted for re-analysis. Manual integrations should be kept to a minimum and should be dated and initialed by the analyst. To determine if analytical results are acceptable, a qualified and independent QA/QC analyst shall conduct a 100% secondary review prior to reporting of the final data package to the client. The laboratory QA/QC Checklist (Attachment 1) is used to indicate any problems with the referenced analytical batches and submitted to the secondary reviewer. The QA/QC checklist addresses any corrective action taken in the laboratory such as dilutions and reanalyses. All calibrations, calculations, and transcriptions are checked for accuracy and QC sample results are evaluated against specified limits by the secondary reviewer and documented on the Secondary Reviewer Checklist (Attachment I). If instrument calibration and recoveries of all QC samples are within the specified criteria, then the data reports will be submitted to the Project Manager as final results with no qualifiers. If recoveries of any QC samples exceed specified limits and re-analysis is not an option, then the samples will be qualified with a Q-flag or as estimated with a "J" qualifier (J= The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.) depending on the corrective action or the QC element that failed. Data will not be reported if significant QC issues affect the batch analyses. Any qualifications or actions taken will be noted in the case narrative.

12.0 POLLUTION PREVENTION & WASTE MANAGEMENT

Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The United States Environmental Protection Agency (USEPA) has established a prevention hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

For information about pollution prevention that may be applicable to laboratories and research institutions, consult "Less is Better: Laboratory Chemical Management for Waste Reduction", available from the American Chemical Society's Department of Government Regulations and Science Policy, 1155 16th Street N.W., Washington, D.C. 20036; (202) 872-4477.

The USEPA requires that laboratory waste management practices conducted be consistent with all applicable rules and regulations. Excess reagents, samples, and method process wastes should be characterized and disposed of in an acceptable manner. The Agency urges laboratories to protect the air, water and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management consult the "Waste Management Manual for Laboratory Personnel", available from the American Chemical Society.

13.0 REFERENCES

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14.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

Attachment 1: Sample GC Laboratory QA/QC Checklist

Table 1: Analyte Details – LOQ/QLs and MS/MSD Spike values shown are for SIM Configuration

Analyte	CAS #	**Minimum RRF	Maximum %RSD	LOQ/QL Water (ug/L)	LOQ/QL Air (ppbv/v)	LOQ/QL Soil (ug/Kg)
Chloromethane	74-87-3	0.01	20%	5	1-50	120
Vinyl Chloride	75-01-4	0.01	20%	2	1-50	80
Chloroethane	75-00-3	0.1	20%	2	1-50	80
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	0.1	20%	2	1-50	80
1,1-Dichloroethene	75-35-4	0.01	20%	2	1-50	80
+Carbon Disulfide	75-15-0	0.1	20%	2	1-50	80
+Methylene Chloride	75-90-2	0.01	20%	2	1-50	80
+t-Butyl-methyl ether	1634-04-4	0.1	20%	2	1-50	80
trans-1,2-Dichloroethene	156-60-5	0.1	20%	2	1-50	80
1,1-Dichloroethane	75-34-3	0.1	20%	2	1-50	80
cis-1,2-Dichloroethene	156-59-2	0.1	20%	2	1-50	80
*+2-Butanone	78-93-3	0.1	20%	50	1-50	2000
Chloroform	67-66-3	0.1	20%	2	1-50	80
1,1,1-Trichloroethane	71-55-6	0.1	20%	2	1-50	80
Cylcohexane	110-82-7	0.1	20%	2	1-50	80
Carbon Tetrachloride	56-23-5	0.1	20%	2	1-50	80
Benzene	71-43-2	0.1	20%	2	1-50	80
1,2-Dichloroethane	107-06-2	0.01	20%	2	1-50	80
Trichloroethene	79-01-6	0.1	20%	2	1-50	80
Methyl Cyclohexane	108-87-2	0.1	20%	2	1-50	80
1,2-Dichloropropane	78-87-5	0.1	20%	2	1-50	80
Bromodichloromethane	75-27-4	0.1	20%	2	1-50	80
cis-1,3-Dichloropropene	10061-01-5	0.1	20%	2	1-50	80
*+4-Methyl-2-pentanone	108-10-1	0.1	20%	50	1-50	2000
Toluene	108-88-3	0.1	20%	2	1-50	80
Trans-1,3-Dichloropropene	10061-02-6	0.1	20%	2	1-50	80
1,1,2-Trichloroethane	79-00-5	0.1	20%	2	1-50	80
Tetrachloroethene	127-18-4	0.1	20%	2	1-50	80
+2-Hexanone	591-78-6	0.1	20%	50	1-50	2000
Dibromochloromethane	124-48-1	0.1	20%	2	1-50	80
1,2-Dibromoethane	106-93-4	0.1	20%	2	1-50	80
Chlorobenzene	108-90-7	0.1	20%	2	1-50	80
Ethylbenzene	100-41-4	0.1	20%	2	1-50	80
M+p-Xylenes	108-38-3 106-42-3	0.1	20%	2	1-50	80
o-Xylene	95-47-6	0.1	20%	2	1-50	80

Analyte	CAS #	**Minimum RRF	Maximum %RSD	LOQ/QL Water (ug/L)	LOQ/QL Air (ppbv/v)	LOQ/QL Soil (ug/Kg)
Styrene	100-42-5	0.1	20%	2	1-50	80
Bromoform	75-252	0.1	20%	2	1-50	80
Isopropylbenzene	98-82-8	0.1	20%	2	1-50	80
Bromofluorobenzene	460-00-4	0.1	20%	2	1-50	80
1,1,2,2-Tetrachloroethane	79-34-5	0.1	20%	2	1-50	80
1,3-Dichlorobenzene	541-73-1	0.1	20%	2	1-50	80
1,4-Dichlorobenzene	106-46-7	0.1	20%	2	1-50	80
1,2-Dichlorobenzene	95-50-1	0.1	20%	2	1-50	80
+1,2-Dibromo-3-chloropropane	96-12-8	0.1	20%	2	1-50	80
1,2,4-Trichlorobenzene	120-82-1	0.1	20%	2	1-50	80
1,3,5-Trimethylbenzene	108-67-8	0.1	20%	2	1-50	80
1,2,4-Trimethylbenzene	95-63-6	0.1	20%	2	1-50	80
Naphthalene	91-20-3	0.1	20%	2	1-50	80

*Ketones or other VOC compounds not listed here may be analyzed for as verified by a successful method verification study. Method validations are performed at time of analysis when other compounds outside this list are added for a project.
**Minimum response factors for a select number of compounds are lower than what is recommended in 8260C due to the fact that this method deploys solid phase microextraction as opposed to purge and trap and these minimum RFs are acceptable as demonstrated with demonstration of an acceptable LOQ standard.

+- Poor Performer

Table 2: LCS Control Limits for DoD Projects (presented in terms of percent recovery)

Analyte	CAS #	Lower DoD Control Limit (Water)	Upper DoD Control Limit (Water)	Lower DoD Control Limit (Solids)	Upper DoD Control Limit (Solids)
Chloromethane	74-87-3	40	125	50	130
Vinyl Chloride	75-01-4	50	145	60	125
Chloroethane	75-00-3	60	135	40	155
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	NA	NA	NA	NA
1,1-Dichloroethene	75-35-4	70	130	65	135
Carbon Disulfide	75-15-0	35	160	45	160
Methylene Chloride	75-90-2	55	140	55	140
t-Butyl-methyl ether	1634-04-4	65	125	NA	NA
trans-1,2-Dichloroethene	156-60-5	60	140	65	135
1,1-Dichloroethane	75-34-3	70	135	75	125
cis-1,2-Dichloroethene	156-59-2	70	125	65	125
2-Butanone	78-93-3	30	150	30	160
Chloroform	67-66-3	65	135	70	125
1,1,1-Trichloroethane	71-55-6	65	130	70	135
Cylcohexane	110-82-7	NA	NA	NA	NA
Carbon Tetrachloride	56-23-5	65	140	65	135
Benzene	71-43-2	80	120	75	125
1,2-Dichloroethane	107-06-2	70	130	70	135
Trichloroethene	79-01-6	70	125	75	125
Methyl Cyclohexane	108-87-2	NA	NA	NA	NA
1,2-Dichloropropane	78-87-5	75	125	70	120
Bromodichloromethane	75-27-4	75	120	70	130
cis-1,3-Dichloropropene	10061-01-5	70	130	70	125
4-Methyl-2-pentanone	108-10-1	60	135	45	145
Toluene	108-88-3	75	120	70	125
Trans-1,3-Dichloropropene	10061-02-6	55	140	65	125
1,1,2-Trichloroethane	79-00-5	75	125	60	125
Tetrachloroethene	127-18-4	45	150	65	140
2-Hexanone	591-78-6	55	130	45	145
Dibromochloromethane	124-48-1	60	135	65	130
1,2-Dibromoethane	106-93-4	80	120	70	125
Chlorobenzene	108-90-7	80	120	75	125
Ethylbenzene	100-41-4	75	125	75	125
M+p-Xylenes	108-38-3 106-42-3	75	130	80	125
o-Xylene	95-47-6	80	120	75	125
Styrene	100-42-5	65	135	75	125
Bromoform	75-252	70	130	55	135
Isopropylbenzene	98-82-8	75	125	75	130
1,1,2,2-Tetrachloroethane	79-34-5	65	130	55	130
1,3-Dichlorobenzene	541-73-1	75	125	70	125
1,4-Dichlorobenzene	106-46-7	75	125	70	125

Analyte	CAS #	Lower DoD Control Limit (Water)	Upper DoD Control Limit (Water)	Lower DoD Control Limit (Solids)	Upper DoD Control Limit (Solids)
1,2-Dichlorobenzene	95-50-1	70	120	75	120
1,2-Dibromo-3-chloropropane	96-12-8	50	130	40	135
1,2,4-Trichlorobenzene	120-82-1	65	135	65	130

Table 3: Example Standard Preparation Calculation Sheet

STONE ENVIRONMENTAL INC

Gas Chromatography Laboratory

HP-5

Standard Prep Log

08/16/05-a1					dmkc	
FID WS						
100	ppm					
x: 8/31/05						
Compound	Vendor	Lot	Exp	Conc (mg/ml)	Amount	
Custom VOC	S	LB18097	8/16/2006	20	20	ul
1,1-DCA	A	B2090306	6/22/2006	20	20	ul
1,1,1-TCA	A	B3050086	6/22/2006	10	40	ul
8020 Mix	A	B1070292	8/31/2005	2	200	ul
1,1-DCE	A	B4050042	1/21/2006	20	20	ul
BFB	A	B4110093	5/17/2006	25	16	ul
MeOH	OmniSolv	44090	3/1/2008	into	3684	ul
				Total	4000	ul

15.0 AUTHORIZATION

Authored by: Kim Watson for Date: 11-7-12

David Crosby, Project Scientist

Approved by: Chris Stone PDC Date: 11-7-12

Seth Pitkin, Vice President

16.0 REVISIONS

Revision 1:

1. Included the analytical process to analyze air in a tedlar bag using SPME and SW846 Method 8260.

Revision 2:

1. Allow a range for the concentration of the internal standard from 8.33 to 16.67 ppb.
2. Section 9.4. Included text to allow for the BFB tune evaluation to be performed as part of a check standard prior to running calibration standards and environmental samples.
3. Table 1. Adjust chloromethane and bromomethane reporting limits to 10 ppb for waters and 120 ppb for soils.
4. Include calibration by linear regression in Section 11.9.

Revision 3:

1. Adjust frequency for continuing calibration check standard from one per 15 analytical runs to one at the beginning of each analytical day.
2. Adjustment of reporting limits to reflect latest method detection limit study.

Revision 4:

1. Add reference to SIM mode analysis on the GC/MS in Sections 2.0, 3.0, and 9.4.2., and definition under Section 3.0.
2. Change EPA to USEPA were applicable.
3. Update typical GC Program parameters in Section 9.1
4. Update QC recovery limits for LCS/ICV from 70-130% to 70-130% and 70-160% for poor performers wherever applicable (Section 11.0) and in Attachment I.
5. Update SS/IS spike concentrations to be an allowed range.
6. Add specifics regarding standard preparation procedures in Section 7.0 referencing the SEI-4.7.5 SOP

and added Table 2 Example Standard Preparation Calculation Worksheet in Section 14.0.

7. Added two new paragraphs under Section 8.2 Sample Handling for pH determinations and percent moisture determination.

Revision 5:

1. Take out reference to an LCS in section 9.4.
2. Add clarification to the LCS Section 9.4.7 regarding the preparation and analysis of soils.
3. Change Section 11.0 to read Section 11.6 ICV, Section 11.7 LCS and 11.8 MS/MSD.

Revision 6:

1. Section 6.0: add SPME Autosampler: CTC Combi PAL.
2. Section 9: Add references for DOD exceptions and references
3. Section 11: Add reference to DOD projects and blank requirements.
4. Add Table 2. Reference to DOD project limits and allowances.

Revision 7:

1. Section 8: Add text requiring unique labeling of sample vials when more than one vial is presented for a sample.

Revision 8:

1. Changed document to reflect SW846 USEPA Method 8260C from 8260B
2. Included updates for DOD exceptions.
3. Minor grammatical and typographical errors fixed.
4. Updated extractions times to include 6 minutes for faster methods.
5. Updated columns use to RTX-VMS
6. Correlation Coefficient of 0.99 is now acceptable for calibrations.
7. Added Surrogate Recovery and Internal Standard Verification to Corrective Action Table
8. Added text for IC/LCS to CA Table.
9. Removed references to CCC's and SPCC's which are not used in method 8260C

Updated QA/QC Checklist added Table 2.

Revision 9:

1. Updated section to 8.2 to include isolation protocol for suspected hot samples.
2. Added LOD, LOQ, and Measurement of Uncertainty
3. Added equipment maintenance

4. Add Secondary Data Review Checklist demonstrating 100% review of all data sets.

Attachment I

SAMPLE GC LABORATORY QA/QC CHECKLIST AND SECONDARY REVIEW CHECKLIST

GC/MS LABORATORY QA/QC CHECKLIST

Instrument:_____ QC Batch:_____

System:_____ VSTD FB Area:_____

Date:_____ VSTD CB(D5) Area:_____

QC Analysis	**Criteria**	**Filename(s):**
IC Calibration	%RSD ≤ 20%, correlation coefficient >0.99 (other)	
SPME Fiber Blank	< 1/2 reporting limit	
BFB Tune Check	per 8260 Method	
CC Calibration	%D ±20% VOCs, ±30% (other)	
Method Blank	< 1/2 reporting limit	
ICV Standard	See Table 2	
MS/MSD Analysis	See Table 2	
Surrogate Standards	70-130% waters 60-140% soils	

Note: DOD projects may require additional and/or different QC criteria then specified above, see pertinent sections of SOP for details.

Comments/Corrective Action:

Prepared By: _____

Reviewed By:_____

SECONDARY DATA REVIEWER CHECKLIST

Instrument:_____

Project ID:_____

System:_____

Sample Delivery Group. (SDG):_____

SDG Date Range:_____

Stone Project Number:_____

Action	**Completed?**
COCs: • Check that all COCs have been properly populated with all necessary information.	
Accuracy & Completeness: • Export and format LIMS tblSampleLogin for inclusion in lab data report, including a header that shows project and SDG info. • Check each sample on this Sample Login Summary against project COCs for accuracy and completeness. • Generate a list of samples not analyzed, including extra vials not opened, for includsion in lab data report • Use LIMS Completeness check ("Samples Logged in/Count of Results" button) to make sure all samples have data in LIMS	
Sample Weights & Percent Moisture: • If soil or rock samples are present in SDG, generate, export and format LIMS "Review Sample Weights" query for inclusion in lab data report, including a header that shows project and SDG info. This is also a handy sheet to use when reviewing results and checking calculations.	
QC Data Review: • ICAL - Check response factor calculations for ICAL (esp. those with manual integrations) and make sure all levels are represented correctly on form – add 7th level RFs if applicable. Make sure that cal ranges are correct in LIMS. • Review all QC data from each analytical batch in the SDG, making sure that all QC Data meet the requirements described on Daily QC/QC Checklist. This review should include the following: FBLK, BFB Tune (including 12-h window), ICAL, CCV, VBLK, LCS, MS/MSD, FD, ISTD and SS recoveries. Sign Daily QA/QC Checklist. • Make sure that all QC outages are documented on Analyst's Daily QA/QC Checklists. If any data qualifications are necessary based on QC outages, confirm that those qualifications have been applied to the proper samples and analytes in tblFinalResults – use filtering tool to search.	

Sample Data Review: • Make sure all results in LIMS (or on LIMS-generated Form 1 Reports) match those on chromatograms, including checking any sample weight and/or percent moisture calculations for soil/rock samples. • Make sure chromatograms say "QT reviewed" and have been signed and dated by analyst. • Check that ISTD and SS are within acceptable range for all samples in window. • Check that all results have been reported from within cal range • Check that qualifiers have been applied appropriately if necessary. • Initial and date chromatogram, as secondary reviewer	
Manual Integrations (For DoD Projects only): • Check all manual integrations by looking at before and after integrations on pdf files. • Verify that analyst has initialed and dated each manual integration. Initial and date next to analyst's initials.	
Lab Narrative Report: • Using the Daily QC/QC Checklists, summarize in narrative any QC outages from the batches within the SDG as well as any problems encountered with samples. • Compile all necessary attachments (dependent of level of reporting). At a minimum, this would include scanned COCs, LIMS Sample Login Summary, Sample weights and percent moisture data (for soil/rock data), and sample Form 1 Reports. • Send final report to another analyst or Lab or QA Manager for review.	
EDD: • Generate EQUIS EDD (or other appropriate format) in LIMS. • Make sure that all columns are properly populated. • Make sure that EDD contains the correct number of results • Spot check results in the EDD against verified Form 1 Reports.	

Secondary Reviewer Signature: _____ Date: _____

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We appreciate your interest in Microbial Insights' CENSUS® analysis. CENSUS®, a molecular biological tool employing quantitative polymerase chain reaction (qPCR), allows site managers to cost-effectively detect and quantify specific microorganisms or functional genes deemed critical for successful bioremediation. Currently, Microbial Insights offers over 30 different CENSUS® assays for assessing biodegradation of a broad spectrum of contaminants ranging from petroleum hydrocarbons to chlorinated solvents.

We would like to take this opportunity to provide some background information for specific CENSUS® assays along with the Cost Proposal requested.

- ***Dehalcoccoides* (qDHC)**: The only bacterial group isolated to date that is capable of complete reductive dechlorination of PCE and TCE to ethene. In fact, the presence of *Dehalococcoides* spp. has been associated with the full dechlorination to ethene at sites across North America and Europe (1).

Elevated *Dehalococcoides* concentrations (≥10^4 Dehalococcoides cells/mL) are associated with complete reductive dechlorination, but the range of chlorinated ethenes metabolized and cometabolized varies by species and strains within the *Dehalococcoides* genus. Since the accumulation of daughter products termed a DCE or vinyl chloride "stall" can be a concern, Microbial Insights offers a group of CENSUS® assays for quantification of *Dehalococcoides* functional genes encoding reductive dehalogenases to more definitively confirm the potential for reductive dechlorination of TCE, *cis*-DCE, and most importantly, vinyl chloride.

- **TCE Reductase (*tce*A):** The *tce*A gene encodes the enzyme responsible for reductive dechlorination of TCE to *cis*-DCE in some strains of *Dehalococcoides*. Although the *tce*A gene is not universally distributed among all strains of *Dehalococcoides* and the absence of *tce*A does not preclude the potential for reductive dechlorination of TCE in the field, detection of the *tce*A gene provides an additional line of evidence indicating the potential for dechlorination of TCE.
- **Vinyl Chloride Reductase (*bvc*A):** The *bvc*A gene encodes the vinyl chloride reductase enzyme responsible for reductive dechlorination of vinyl chloride to ethene by *Dehalococcoides* sp. str. BAV1 (2). While reports of detection frequency vary in the literature, *bvc*A is the only vinyl chloride reductase gene detected at some sites (3) and has been demonstrated as the most abundant and actively expressed vinyl chloride reductase gene in PCE column studies (4). In an internal study, ethene production was observed in 80% of the samples in which the *Dehalococcoides* population was greater than or equal to 10^4 cells/mL. The *bvc*A vinyl chloride reductase gene was detected in over 50% of these samples thus confirming the importance of the *bvc*A vinyl chloride reductase in the complete reductive dechlorination.
- **Vinyl Chloride Reductase (*vcr*A):** The *vcr*A gene encodes the vinyl chloride reductase enzyme responsible for reductive dechlorination of *cis*-DCE and vinyl chloride by *Dehalococcoides* sp. strain VS (5). As with the *bvc*A vinyl chloride reductase gene, detection of the *vcr*A gene is associated with ethene production in internal studies (67%) and vinyl chloride reduction in independent studies (3, 5).

Microbial Insights is a molecular microbiology laboratory – we do not offer commercial bioaugmentation cultures or remediation products. We believe that each client deserves unbiased results that are both accurate and precise. Furthermore, we recognize the value of every client. No matter how large or small your project, we are committed to serving you at each step in the process, from assay selection through data interpretation.

We look forward to working with you.

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Recommended Practices for Core Analysis

RECOMMENDED PRACTICE 40
SECOND EDITION, FEBRUARY 1998

American
Petroleum
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- To work with others to resolve problems created by handling and disposal of hazardous substances from our operations.
- To participate with government and others in creating responsible laws, regulations and standards to safeguard the community, workplace and environment.
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Recommended Practices for Core Analysis

Exploration and Production Department

RECOMMENDED PRACTICE 40
SECOND EDITION, FEBRUARY 1998

American Petroleum Institute

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SCOPE

These recommended practices for core analysis replace API RP 40, *Recommended Practice for Core Analysis Procedure*, 1960, and API RP 27, *Recommended Practice for Determining Permeability of Porous Media*, 1952, (reissued 1956). In the first section of the new recommended practices, Planning a Coring Program, key factors to be taken into consideration in obtaining core samples are explained and advantages of different coring procedures are given. The second section, Wellsite Core Handling Procedures and Preservation, addresses documentation of coring conditions and how cores should be handled once they reach the surface, including marking and preservation. The third section, Core Screening and Core Preparation, describes how the condition and nature of core samples can be documented through core gamma logs and various imaging techniques, and how samples should be selected and prepared for basic testing. Also covered in the third section are methods of preserving samples prior to testing, and procedures for cleaning and drying samples. The fourth section, Fluid Saturation, explains how fluid saturations can be determined on different types of samples and the limitations of the various techniques. The fifth section, Porosity Determination, defines different types of porosity and explains the measurements. The sixth section, Permeability Determination, explains the theory and methods for measurement of permeability of porous media to a single phase. Relative permeability measurements to two or three phases are not covered in this document. The seventh section, Supplementary Tests, covers determination of grain size, brine salinity, oil gravity, and acid solubility. The eighth and final section, Reporting, supplies forms to assist in recording the details of core handling and testing methodology that could be critical in interpreting basic core analysis data.

Core analysis, like other technical areas, is continually evolving so that both methodology and costs are changing. The recommended practices provided here represent a snap shot in time of the consensus advice of a large international committee.

Geologic materials come in a vast range of chemical compositions and physical states. For unusual samples or extraordinarily accurate data, it may be necessary to develop special procedures.

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SECTION 1—PLANNING A CORING PROGRAM

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Recommended Practices for Core Analysis

1 Planning a Coring Program

1.1 GENERAL

1.1.1 Scope

This section addresses the complexities of planning a coring program, the decisions to be made, and the factors that influence the choices.

1.1.2 Principle

A coring program is similar to many engineering projects. It begins with the premise that an investment will reap a reward. It progresses through a phase of exploring alternate sources of information; well tests, logs, previous cores, and cuttings or sidewall cores.

Planning begins by listing the objectives of the coring program. This is best done by a team of petrophysical, reservoir, geological, drilling, and production personnel. When discussing objectives, every expenditure must ultimately lead to producing more oil or gas at a lower unit cost. Constraints in budget, location, and timing will be placed on the program. Hole size, hole angle, temperature, pressure, and rock type will influence the selection of the coring tools. Planning becomes an interactive process where consensus is built and a detailed program formulated.

The keys to a successful coring operation are planning and communication.

1.1.3 Objective

The objective of every coring operation is to gather information that leads to more efficient oil or gas production. Some specific tasks might include the:

a. Geologic objectives:
   1. Lithologic information:
      (a) Rock type.
      (b) Depositional environment.
      (c) Pore type.
      (d) Mineralogy/geochemistry.
   2. Geologic maps.
   3. Fracture orientation.

b. Petrophysical and reservoir engineering:
   1. Permeability information:
      (a) Permeability/porosity correlation.
      (b) Relative permeability.
   2. Capillary pressure data.
   3. Data for refining log calculations:
      (a) Electrical properties.
      (b) Grain density.
      (c) Core gamma log.
      (d) Mineralogy and cation exchange capacity.
   4. Enhanced oil recovery studies.
   5. Reserves estimate:
      (a) Porosity.
      (b) Fluid saturations.

c. Drilling and completions:
   1. Fluid/formation compatibility studies.
   2. Grain size data for gravel pack design.
   3. Rock mechanics data.

1.1.4 Coring Fluids

1.1.4.1 The selection of a coring fluid should be based on four points:

a. Safety.
b. The primary objective of the coring program.
c. Environmental concerns.
d. Cost.

1.1.4.2 Safety takes precedence over all other factors. The drilling fluid must be designed to hold the expected formation pressures as well as clean, lubricate, and stabilize the borehole. The objectives of the coring program should influence the selection of the coring/drilling fluid. All coring fluids should be designed to have low static API filter loss and very low dynamic spurt loss to minimize core flushing.

1.1.4.3 Environmental concerns should also be considered and budgeted for. This may mean using a more expensive drilling fluid system to meet environmental objectives, or providing additional drilling fluid handling equipment to ensure containment.

1.1.4.4 Cost is important; still, it is a good practice to review the cost of the entire core analysis program and the expected benefits from it while pricing drilling fluid systems. Savings on drilling fluids may increase the cost of the core analyses, and put the accuracy of the core studies at risk.

1.1.4.5 The question of which drilling fluid is best for coring cannot be answered directly. Water-based, oil-based, foam, and air/mist drilling fluids have all been used to successfully cut cores. The best recommendation is to follow the criteria given above. Evaluating the needs of the drilling and core analysis program will lead to an appropriate selection.

1.2 CORING EQUIPMENT

1.2.1 Scope

This section presents an overview of coring tools, including guidelines for selecting coring tools for specific applications. Details of particular coring systems, and job specific coring recommendations should be obtained from appropriate service companies.

1.2.2 Principle

Coring equipment is designed to retrieve rock samples from deep in the earth for geologic and engineering studies. The tools do an excellent job of recovering core material, and specialized equipment has been developed to trap reservoir fluids and even seal in bottom-hole pressure.

1.2.3 Apparatus

With several notable exceptions coring systems consist of an inner core barrel suspended by a swivel assembly within an outer core barrel that is attached to the drill string. A coring bit is attached to the bottom of the outer barrel and a core catcher is fitted to the bottom of the inner core barrel. Drilling fluid is pumped down the drill string, through the swivel assembly, through the annulus between the inner and outer core barrels, and out the core bit.

1.3 CONVENTIONAL CORING SYSTEMS

1.3.1 Conventional Core Barrel

Conventional coring tools are available to cut cores with outer diameters from 1.75 to 5.25 inches (44.5 to 133.4 millimeters). Core length can run from 1.5 feet (.46 meter) for short radius horizontal well applications to over 400 feet (121.9 meters) for thick, uniform, consolidated formations. Hole size, hole angle, rock strength, and lithology will control the diameter and length of core that may be cut in one trip. The final selection of a particular system will depend upon the formation, location, and objectives of the coring program. Table 1-1 summarizes the conventional coring options available.

1.3.2 Heavy-Duty Conventional Core Barrels

Special heavy-duty coring tools have been developed to core harder than normal formations, and cut extended length cores. Heavy duty threads allow more torque to be applied to the bit, and improve the margin of safety against tool failure. Designed to cut cores up to 5.25 inches (133.4 millimeters) in diameter, these tools are especially attractive in situations where rig time is the largest coring expense. Heavy-duty coring systems are used to best advantage when coring longer lengths of homogeneous formations or when anticipating higher than normal torque loads.

The marine core barrel was the precursor to today's generation of heavy-duty core barrels. Developed to be stronger than existing coring systems, the tool was developed for use in offshore applications. The marine core barrel does increase the margin of safety against tool failure, but is restricted to cutting a 3-inch (76.2-millimeter) diameter core.

1.3.3 Core Barrel Liners

The use of a core barrel liner in a steel inner core barrel has two primary functions: to improve core quality by physically supporting the core material during handling and to serve as a core preservation system. PVC and ABS plastic, fiberglass, and aluminum have all been used as inner core barrel liners. The liners slip inside a conventional inner core barrel and are held in place by the core-catcher assembly and friction. Liners are typically 30 feet (9.14 meters) long. They may be cut shorter for special applications, but their maximum length is rarely more than 30 feet (9.14 meters) due to manufacturing and material handling limitations.

Liners are most often specified when coring unconsolidated or fractured formations. They are also appropriate when cutting hard rock in remote and offshore locations when immediate core preservation is required. Plastic liners are suitable up to temperatures of 180°F (82.2°C). Fiberglass liners may be used up to 250°F (121°C); 350°F (176.7°C) if special high temperature resin is used. Aluminum is generally recommended when temperatures in excess of 250°F (121°C) are expected. The disadvantage of core barrel liners is that

Table 1-1—Conventional Coring Systems

Inner Barrel	Core Length	Special Features
Mild steel	30 to 120 ft. (9.14 to 36.58 m)	Ready-made core preservation system. High temperature applications.
Mild steel	1.5 ft. (.46 m)	Designed for short-radius coring.
High strength steel	120 to >400 ft. (36.38 to >121.9 m)	Stronger barrel, includes additional inner and outer core barrel stabilization.
Fiberglass	30 to 90 ft. (9.14 to 27.43 m)	Ready-made core preservation system. Used for consolidated and unconsolidated formations. Maximum operating temperatures: normal resin 250°F (121°C), high temperature resin 350°F (176.7°C).
Aluminum	30 to 90 ft. (9.14 to 27.43 m)	Ready-made core preservation system. High temperature applications, maximum 350°F (176.7°C).
Steel with a plastic liner	30 ft. (9.14 m)	Ready-made core preservation system. Maximum temperature of 180°F (82.2°C). Reduces core diameter by $^1/_2$ in. (12.7 mm).
Steel with a fiberglass liner	30 ft. (9.14 m)	Ready-made core preservation system. Maximum temperature of 250°F (121°C). Reduces core diameter by $^1/_2$ in. (12.7 mm).
Steel with an aluminum liner	30 ft. (9.14 m)	Ready-made core preservation system. Maximum temperature of 350°F (176.7°C). Reduces core diameter by $^1/_2$ in. (12.7 mm).

they reduce the effective diameter of the inner core barrel by approximately 0.5 inch (12.7 millimeters).

1.3.4 Disposable Inner Core Barrels

Disposable inner core barrels serve the same general purposes as core barrel liners. They improve core quality by physically supporting the core material during handling and serve as a core preservation system. In addition, the outside diameter of the core is not reduced, as it would be with an inner barrel liner. Disposable inner core barrels are available in aluminum, fiberglass, and mild steel, and are manufactured in a variety of sizes to fit most conventional coring systems. In addition, the fiberglass inner core barrel has a low coefficient of friction that allows the core to slide more easily into the core barrel, thereby reducing the risk of core jamming.

1.3.5 Coring High Angle or Horizontal Well

Medium radius [290 to 700 feet (88.4 to 213.4 meters) radius] and extended length wells can be cored with conventional core barrels powered from the rotary table or by a downhole motor. Most cores will be cut without using a downhole motor, but cases will arise where the use of a mud motor is justified. Using a downhole motor enables coring to proceed without rotating the drill string. Typically a 30-foot (9.14-meter) long conventional core barrel would be placed ahead of the downhole mud motor. Mud motors produce high torque at low rotating speed for optimum coring power. Core barrel length and core diameter may be varied to accommodate drilling constraints. The inner core barrel is stabilized by fitting it with special roller bearing or bushing assemblies to centralize the inner core barrel. A special drop ball sub may be placed between the motor and core barrel to allow drilling fluid to flow through the inner core barrel, cleaning it of debris before coring. Activating the sub diverts the drilling fluid flow between the inner and outer core barrel for coring.

In some instances during coring it may be necessary to keep very tight control on the angle of the well. Coring without the downhole motor may improve well-angle control.

1.4 SPECIAL CORING SYSTEMS

1.4.1 General

Special coring systems have evolved to fill specific coring needs. Pressure-retained and sponge core barrels arose from a need for better oil saturation data. The rubber-sleeve and full-closure coring systems were developed specifically to improve the quality of cores cut from unconsolidated formations. Other special coring systems have equally unique capabilities, making them all useful to the engineers and geologists employing them. Table 1-2 summarizes some of the available special coring options.

1.4.2 Pressure-Retained Coring

Pressure-retaining core barrels are designed to retrieve cores maintained at reservoir pressure conditions. Accepted as the best method for obtaining core-based oil saturation data, pressure-retained cores also capture reservoir gases. The tool is especially useful when studying the feasibility of enhanced recovery projects and estimating the methane content of coal.

Pressure-retained core barrels are available in two sizes: 6-inch (152.4-millimeter) and 8-inch (203.2-millimeter) outside diameter that cut cores 2.50- and 3.75-inch (63.5- and 95.3-millimeter) outside diameter, respectively. The 6-inch (152.4-millimeter) outside diameter barrel cuts up to 20 feet (6.1 meters) of 2.5-inch (63.5-millimeter) diameter core while holding a maximum of 10,000 psi (69 MPa) pressure. The 8-inch (203.2-millimeter) outside diameter barrel cuts 10 feet (3.05 meters) of 3.75-inch (95.3-millimeter) diameter core while retaining a maximum of 5,000 psi (34.5 MPa) internal pressure. The maximum recommended operating temperature is 180°F (82°C).

Pressure core barrels are sophisticated tools requiring an on-site facility to service the barrel and handle the pressurized cores. Core handling procedures may be found in 2.2.5.

Table 1-2—Special Coring Systems

Coring System	Maximum Core Dimensions	Special Applications
Pressure-retained	3.75 in. x 10 ft. (5000 psi) [95.3 mm x 3.05 m (34.5 MPa)] 2.5 in. x 20 ft. (10000 psi) [63.5 mm x 6.1 m (69 MPa)]	Pressure-retained analyses, fluid saturations, gas volume and composition.
Sponge-lined	3.5 in. x 30 ft. (88.9 mm x 9.1 m)	Fluid saturations.
Full-closure	4.0 in. x 60 ft. (101.6 mm x 18.3 m)	Recovering unconsolidated formations.
Rubber-sleeve	3.0 in. x 20 ft. (76.2 mm x 6.1 m)	Recovering unconsolidated, fractured, or conglomeritic formations.
Wireline retrievable	2.75 in. x 30 ft. (69.9 mm x 9.1 m)	Coring is possible without tripping pipe.
Wireline percussion sidewall	1 in. x 1.75 in. (25.4 mm x 44.5 mm)	Samples obtained after drilling and logging.
Wireline drilled sidewall	.94 in. x 1.75 in. (23.9 mm x 44.5 mm)	Samples obtained after drilling and logging.
Sidewall corer	2.5 in. x 10 ft. (63.5 mm x 3.05 m)	Core obtained after drilling and logging.

1.4.3 Sponge-Lined Coring System

The sponge-lined coring system was developed to improve the accuracy of core-based oil saturation data. A sponge coring system does not trap reservoir gases, instead it traps oil expelled as the core is brought to the surface. The saturation information is very useful when evaluating enhanced oil recovery projects.

A sponge coring system has the advantage of being less expensive to operate than a pressure-retained coring system, while providing an opportunity to improve the accuracy of the core based oil saturation data. The sponge is stable to a temperature of 350°F (176.7°C). The sponge coring system is limited to cutting a maximum of 30 feet (9.14 meters) of 3.5-inch (88.9-millimeter) diameter core per trip.

1.4.4 Full-Closure Coring Systems

Full-closure coring systems were developed to improve the recovery of unconsolidated formations. These systems use core barrel liners or disposable inner core barrels, and a special core catching system to retrieve the troublesome rocks.

Full-closure coring technology allows the inner core barrel to slip gently over soft core with a minimum of disturbance, and then seal the core within the core barrel. This is done by using a full-closure core catcher assembly that allows unobstructed entry of the core into the inner core barrel, and then after coring seals off the bottom of the inner barrel. Full-closure coring systems are currently limited to cutting either 3.5-inch (88.9-millimeter) or 4-inch (101.6-millimeter) diameter cores. The recommended core length is 30 feet (9.14 meters). The smooth bore and the absence of an exposed core catcher may result in lost core if the tool is lifted off bottom before activating the full-closure core catcher.

1.4.5 Rubber-Sleeve Core Barrel

The rubber-sleeve coring system was the first system developed to improve the chances for recovering unconsolidated sands, conglomerates, and hard fractured formations. The rubber-sleeve barrel is unique in that the top of the inner barrel does not move relative to the core during coring. The outer barrel is drilled down around a column of rock that is progressively encased in a rubber sleeve. The rubber sleeve is smaller than the diameter of the core; it stretches tightly around the core, wrapping it securely and protecting it from the scouring action of the drilling fluid. The core is supported by the rubber sleeve thus, aiding in the recovery of soft formations that would not support their own weight.

There is only one size of rubber-sleeve core barrel, that cuts 20 feet (6.1 meters) of 3-inch (76.2-millimeter) diameter core per trip. The rubber sleeve itself is limited to temperatures no higher than 200°F (93°C). The tool is not recommended for use in holes with more than 45 degrees of inclination. In addition, coring must be stopped approximately every two feet to allow the tool to be reset; this might lead to core jamming in fractured formations. The system works best from fixed drilling structures, yet it can be operated from floating rigs if rig movement is minimal.

1.4.6 Wireline-Retrievable Core Barrel

Wireline-retrievable coring tools are operationally similar to conventional coring systems except they are designed for the inner core barrel to be pulled to the surface by a wireline. This speeds the coring operation by eliminating the need to trip the entire drill string for each core. A new section of inner core barrel is pumped down the drill string and latched into place for additional coring, or a drill plug is pumped down to facilitate drilling ahead.

Wireline-retrievable coring tools are usually smaller and lighter than conventional coring systems. This is an asset when they must be transported to remote locations or by helicopter. Unfortunately, the core diameters are limited since the entire inner core barrel assembly must pass through the drill string. Also, care must be taken to prevent “swabbing” oil or gas into the wellbore as the inner barrel is recovered.

1.5 WIRELINE SIDEWALL CORING

1.5.1 General

Wireline sidewall coring systems were developed to obtain core samples from a wellbore after it has been drilled and logged, and before casing is run. These tools may be positioned in zones of interest using data from gamma or spontaneous potential logs as guides. The samples provide small pieces of formation material, suitable for geologic and engineering studies.

1.5.2 Percussion Sidewall Coring

Most wireline sidewall cores are obtained by percussion sidewall coring systems. These tools shoot hollow, retrievable, cylindrical bullets into the wall of an uncased hole. The tool (gun) is lowered to the desired depth on a wireline, and then fired by electrical impulses controlled from the surface. The bullets remain connected to the gun by wires, and movement of the gun pulls the bullets, containing the samples, from the hole wall. Up to 66 samples, 1 inch (25.4 millimeters) in diameter by $1^{3}/_{4}$ inches (44.5 millimeters) in length, may be taken during one downhole trip. Different bullet “core barrel” designs are available for unconsolidated, soft, and medium-to-hard formations. It is wise to have more than one type of core barrel on location until acceptable core recovery can be shown.

The advantages of percussion sidewall coring are speed, low cost, and the ability to sample zones of interest after open hole logs have been run. The disadvantage is that the bullet usually alters the formation, shattering harder rock or compressing softer sediments. This reduces the quantitative value of the sidewall core analysis data. Percussion sidewall core

recovery tends to be low in very hard or fractured rock, and in very permeable unconsolidated sand.

1.5.3 Drilled Sidewall Coring

The rotary or drilled sidewall coring tool was developed to recover wireline sidewall core samples without the shattering impact of the percussion system. Suitable for hard-to-friable rock, the rotary sidewall coring tool uses a diamond-tipped drill to cut individual samples. Leverage applied to the drill snaps the sample from the sidewall. The drill and sample are retracted into the body of the tool where the sample is deposited. The tool is moved to a new location after depositing each sample. A maximum of 30 samples, $^{15}/_{16}$-inch (23.9 millimeter) diameter by $1^{3}/_{4}$-inch (44.5-millimeter) length, may be taken during one trip.

An advantage of the rotary sidewall coring system is that it produces samples of hard rock suitable for quantitative core analysis. Disadvantages are that it is more expensive than percussion sidewall coring in terms of rig time costs, and sample recovery tends to be low in unconsolidated formations.

1.5.4 Sidewall Coring Systems

Some new sidewall coring systems are coming on the market, and they merit discussion for two reasons. First, they are designed to acquire a larger, more-continuous core sample from a drilled and logged wellbore than is possible with existing sidewall coring tools. Secondly, the emergence of new tools confirms there is still room for improvement in the area of acquiring high quality, low cost core samples.

The first system is similar to a conventional core barrel. The sidewall coring system is designed to cut up to 10 feet (3.05 meters) of $2^{1}/_{2}$-inch (63.5-millimeter) diameter core. The tool is attached to a conventional drill string and lowered to the zone of interest. There an integral arm pushes the core barrel against one side of the wellbore. From then on the tool is operated as a conventional core barrel. The second system uses a removable whipstock to direct a conventional core barrel out into the formation. Both systems address the need to acquire quality core samples after logging.

1.6 ORIENTED CORING

1.6.1 General

Oriented cores are used to orient fractures, stress fields, and permeability trends. Exploration, production, and drilling operations use the information to explore for fractured reservoirs, design waterfloods, and plan horizontal wells.

Oriented cores are typically cut using a conventional core barrel fitted with a special scribe shoe, and a device for recording the orientation of the primary scribe knife relative to magnetic north. Laboratory methods used to orient cores are correlation of the core with borehole imaging logs and the paleomagnetic method. Table 1-3 lists methods commonly used to orient cores.

Table 1-3—Core Orientation Methods

Method	Location	Comments
Multishot survey	Well	Must stop drilling to take reading.
Electronic survey	Well	Records orientation versus time.
Paleomagnetic method	Laboratory	Orients one continuous interval.
Log correlations	Laboratory	Requires correlatable features in core and wellbore.

1.7 CORING BITS

1.7.1 General

Coring bits are a basic part of the coring system. Unfortunately for the experts and novices alike, coring bits come in a bewildering array of styles. Fortunately, general bit/formation guidelines are available from manufacturers to aid in selection of the proper bit. With a little background information, it is possible to make informed decisions on cutter types, bit profile, and hydraulic considerations for the range of anticipated coring conditions. Final bit selection should be guided by the goals of the coring program, coupled with a confirmation that the bit has proven itself in the field for similar applications.

The hardness (compressive strength), abrasivity, and variability of the rocks to be cored will have the greatest influence on cutter selection. General guidelines suggest use of smaller, more impact-resistant cutters as the formations get harder.

Low-invasion, face-discharge core bits designed for unconsolidated-to-medium strength formations can be used in harder or more abrasive rocks, but bit life may be drastically reduced.

The information presented in Table 1-4 provides an overview of the types of coring bits that are available. Specific details on coring bits and recommendations for particular applications should be obtained from service companies.

1.7.2 Natural Diamond Bits

Natural diamond core bits are used when the formation is too hard (high compressive strength) and/or abrasive for other type cutting elements. Large natural diamonds can be surface-set in a tungsten carbide matrix, or fine diamond chips can be dispersed in matrix to form what is called an impregnated diamond bit. Impregnated natural diamond bits are for ultra-hard formation applications.

1.7.3 Polycrystalline Diamond Compact—PDC

Polycrystalline diamond compact (PDC) cutters are man-made diamond materials that consist of a layer of micron sized diamond grit sintered together and bonded to tungsten carbide studs. The thickness of the polycrystalline diamond layer is only 0.020 to 0.060 inches (.51 to 1.52 millimeters). PDC bits are used to efficiently core formations ranging from

Table 1-4—General Coring Bit Guide

Rock Properties	Rock Type	Core Bit
Ultra-hard, abrasive rock	Quartzite, Igneous Rocks	Impregnated natural diamond.
Hard, abrasive rock	Sandstone, Shale, Siltstone	Natural diamonds surface set or TSP cutters.
Hard, non-abrasive rock	Limestone, Dolomite, Anhydrite	TSP cutters.
Medium to hard rock with abrasive layers	Sandstone, Limestone, Shale	TSP or surface set natural diamonds.
Soft to medium strength rock	Sandstone, Chalk, Shale	PDC cutters, low fluid invasion design.
Soft rocks, no sticky layers	Salt, Anhydrite, Shale	PDC or roller cone cutters.
Soft, sticky rock	Gumbo Clay	PDC cutters, face discharge.

very soft to medium hard. The bits are designed to cut by shearing resulting in a rapid rate of penetration. Due to the geometry of the PDC cutter, they are susceptible to impact damage, and therefore, are not recommended for very hard, highly fractured, or cherty formations.

1.7.4 Thermally Stable Diamond—TSP

Thermally stable (diamond) product, TSP, is similar to PDC in that it is also a man-made diamond material. The main difference in the TSP material is that it has a higher range of thermal stability due to the leaching of the metal catalyst used in the sintering process of manufacture. These cutters are suitable for formations generally considered too hard and/or abrasive for PDC cutters. They are not recommended for soft formations.

1.7.5 Roller Cone

The roller cone core bit uses four rotating cones set with tungsten carbide inserts or hard-faced milled-tooth cutters for coring purposes. The cutters in the cones roll and impact the hole bottom and fail the formation in compression by a chipping action. Due to the slow cutting action (chipping-compressive failure) and the number of moving parts, roller cone core bits are not commonly used.

1.8 FLUID DISCHARGE CHARACTERISTIC OF CORE BITS

1.8.1 Throat Discharge

Throat discharge core bits are designed to have 100 percent of the fluid pass between the core shoe and the inside diameter of the core bit (the “throat”). Throat discharge bits are designed to clean the inside diameter of the core bit, removing cuttings from this area to ensure a very smooth entry of the core into the core barrel. The cleaning action reduces the tendency of hard and/or brittle formations to jam.

1.8.2 Face Discharge

Face discharge core bits are designed to divert some fluid that would normally pass through the throat of the bit to the face of the bit. This cleans the face of the bit and reduces the amount of fluid scouring the core as it enters the core barrel. Face discharge bits are recommended for use in soft and friable formations.

1.8.3 Low-Invasion Profile

The low-invasion profile coring bits are designed to maximize penetration rate, and minimize drilling fluid filtrate invasion into the core. The design incorporates face discharge ports, a reduced number of cutters, and a diminished clearance between the inner core barrel and the bit face. Use of low-invasion profile core bits is recommended for soft to medium strength formations. Harder formations would slow the rate of penetration and possibly damage the cutters.

1.9 CORE CATCHERS

1.9.1 General

The single most critical part of every coring system is the core catcher that holds the core in the barrel as it is brought to the surface. Table 1-5 lists the core catchers available and suggests those most appropriate for specific rock types.

Many situations call for combining two or more catchers to ensure success. Sequences of friable sand interbedded with shale might require both slip and flapper type catchers. Full-closure catchers, run primarily to ensure success when coring unconsolidated sand, also incorporate split-ring or slip-type catchers to improve core recovery in case coring ends in hard rock.

Table 1-5—Core Catchers

Type	Recommended Usage
Split-ring, spring	Consolidated formations.
Collet	Where formation characteristics are unknown.
Slip	Consolidated formations, normally run with flapper catcher or with orientation knives.
Dog or flapper	Consolidated, fractured, and unconsolidated formations where geology is unknown.
Basket	Unconsolidated formations, normally run with another core catcher type.
Full closure	Friable to unconsolidated formations to provide positive full closure.

SECTION 2—WELLSITE CORE HANDLING PROCEDURES AND PRESERVATION

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2 Wellsite Core Handling Procedures and Preservation

2.1 GENERAL

2.1.1 The recommendations included in this document may involve the use of hazardous materials, operations, and equipment. This document does not address all of the safety problems associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2.1.2 Wellsite core handling procedures and preservation should follow the best possible practices because the value of all core analysis is limited by this initial operation. The objectives of a core handling program are as follows:

a. Obtain rock material that is representative of the formation.
b. Minimize physical alteration of the rock material during core handling and storage.

The major problems confronting those handling and preserving reservoir rocks for core analysis are as follows:

a. Selection of a nonreactive preservation material and a method to prevent fluid loss or the adsorption of contaminants.
b. Application of appropriate core handling and preservation methods based upon rock type, degree of consolidation, and fluid type.

Different rock types may require additional precautions in obtaining representative core data (see 2.4). All core material should be preserved at the wellsite as soon as possible after retrieval to minimize exposure to atmospheric conditions.

2.1.3 The terminology that has evolved to describe the state of core preservation is important historically, but may be confusing because it is sometimes not used consistently. For example, the term “native state” has been often used to designate core drilled with oil-based mud or lease crude for the purpose of making accurate water saturation measurements. Similarly, “fresh state” has often been used to imply that core was drilled with bland, water-based drilling fluid and preserved at the wellsite to limit evaporative losses. This term has also been used to include cores cut with oil-based mud. In the interests of consistency, the following terminology is recommended:

2.1.3.1 fresh core: Any newly recovered core material preserved as quickly as possible at the wellsite to prevent evaporative losses and exposure to oxygen. The fluid type used for coring should be noted, e.g., fresh state (oil-based drilling fluid), fresh state (water-based drilling fluid).

2.1.3.2 preserved core: Similar to fresh core, but some period of storage is implied. Preserved core is protected from alteration by any of a number of techniques (see 2.5).

2.1.3.3 cleaned core: Core from which the fluids have been removed by solvents. The cleaning process (sequence of solvents, temperature, etc.) should be specified.

2.1.3.4 restored-state core: Core that has been cleaned, then reexposed to reservoir fluids with the intention of reestablishing the reservoir wettability condition. This is often the only alternative available, but there is no guarantee that reservoir wettability is restored. The conditions of exposure to the crude oil, especially initial water saturation, temperature and time, can all affect the ultimate wettability.

2.1.3.5 pressure-retained core: material that has been kept, so far as possible, at the pressure of the reservoir in order to avoid change in the fluid saturations during the recovery process.

None of these terms alone adequately describes the state of the core; a full description of drilling mud, handling, preservation, and subsequent treatment is required.

2.1.4 For testing, the core must be sampled. In order to obtain a representative core analysis from the formation(s) of interest, it is recommended that the entire core be sampled. The entire core section should be retained. Wellsite core sampling can be important for a variety of reasons (see 2.3.1). If sampling of the core is required, it should be performed with an awareness that the sampling procedure may impact future core analysis efforts and results. Wellsite sampling must be minimal to maintain the integrity of the core. Samples for lithology description, for example, can be taken from small broken pieces of core without damaging any of the intact rock. If intact, measurable lengths of core are removed, a note or block should be left in their place describing the sample length, lithology, the reason for removal, and any other pertinent information. If samples are required from within an intact core segment, a non-percussive sampling method should be employed. The objective of a standard core-sampling procedure is to obtain samples under a uniform procedure so that the results will be independent of human bias. The selection of samples is fairly simple for uniform formations. However, where a formation contains widely varying lithology and heterogeneous porosity types (such as conglomerates, cherts, vugular or fractured reservoirs, and interlaminated shales and sands), the proper selection of representative samples requires greater care. A qualified person (engineer, geologist, etc.) should follow an established sampling procedure to minimize statistical bias.

2.1.5 The prescribed core handling and preservation procedures are applicable to all conventionally cored rock material. Many of the same practices apply to sidewall cores and drill cuttings. These recommended procedures have been selected as those which will yield core materials for the most reliable and representative core analysis. The success of any given technique is directly related to the rock properties of the core. Handling procedures should also be based on the technology used to retrieve the rock material and the objectives of the coring program. A review of core preservation materials is also presented. Each coring job and reservoir should be carefully examined prior to the design of a wellsite handling and preservation program.

2.2 CORE HANDLING PROCEDURES

2.2.1 General

Several methods are available for core acquisition. Conventional, full-diameter, continuous coring techniques can be divided into two groups; those that employ a standard reusable steel inner barrel, and those that utilize disposable inner barrels or liners. Other coring methods such as the sidewall devices and wireline-retrieved coring apparatus obtain rock material using special equipment. Special coring processes, including pressure-retained and sponge methods, are available to obtain core and fluid analysis results more representative of in situ conditions.

Consolidated core material obtained with a standard reusable inner barrel should be removed from the barrel as soon as possible after reaching the surface to minimize drilling fluid imbibition. Among the possible undesirable effects of fluid imbibition are as follows:

a. Changes in fluid saturations, geochemical and gas solution equilibrium.

b. Changes in wettability.

c. Mobilization of interstitial clays and fine-grained minerals.

d. Clay swelling and associated degradation of mechanical properties.

Any delay in removal of the core from the barrel should be reported. Various rock types and coring methods require varying levels of attention and can be broken into two major categories:

a. Basic handling—This category requires minimum training and/or experience and includes:

1. Standard reusable steel inner barrel used to obtain core on fairly homogeneous consolidated rock.

2. Wireline sidewall core acquisition from percussion or rotary coring.

b. Special handling—This category requires extensive training and/or equipment and includes:

1. Disposable inner barrels and oriented core barrels used to obtain core from fractured or unconsolidated rock that may require mechanical stabilization (Skopec, et al., 1992).

2. Pressure-retained core barrel to maintain core at reservoir pressure to minimize fluid expansion from pressure reduction and fluid expulsion as the core is brought to the surface (Sattler, et al., 1988).

3. Aluminum core barrel with sponge liner inside a standard steel inner barrel to trap fluids during expansion from pressure reduction as the core is brought to the surface (Park, 1983).

The use of any core inner barrel liner reduces the diameter of the resultant core.

2.2.2 Removal of the Core From a Standard Reusable Steel Inner Barrel

The core should be removed from the inner core barrel in a horizontal position whenever possible. Care should be exercised to minimize the mechanical shock during extraction. The core should be allowed to slide out of the core barrel by slightly elevating the top end of the core barrel. If the core will not slide a rod may be used to push the core from the barrel. It may be necessary to gently tap the core barrel with a hammer to initiate movement of the core. However, do not hammer the core barrel in a manner that imparts severe mechanical shock to the core. In all physical manipulations, attempt to expose the core to the minimum mechanical stress. If the core cannot be removed by the foregoing method, it should be pumped out of the barrel with a fluid. If this is necessary, a suitable piston arrangement should be used that will prevent fluids from directly contacting and contaminating the core. The coring fluid should be used if pumping directly with fluids is necessary. The use of fresh water or other fluids foreign to the core should be avoided. If water is forced past the piston and contacts the core, erroneously high water saturation values may be obtained in subsequent core analysis as any excessive pressuring of the barrel may cause fluid to penetrate the core. Any difficulty or irregularity encountered while removing the core from the barrel should be noted, e.g., pressure used if pumped out with fluid, loss of core material, etc.

2.2.3 Labeling and Logging of the Core

The core should be laid out and boxed on the rig floor if space is available. Alternatively, the pipe rack can be used for this purpose. The labeling and logging of the core should not interfere with the drilling and/or coring operation. If the decision is made to handle the core on the rig floor, place appropriately marked trays, boxes, or troughs near the core barrel. If the core is to be laid out on the catwalk, prepare a cleared area and place the core between two lengths of drill pipe.

Care must be taken to maintain the orientation, and to preserve the correct sequence of the core pieces. The key point is

that the core must be labeled and marked in such a way that the entire cored interval can be reassembled at some future time. The core should be protected from temperature extremes, moisture, and dehydration, i.e., direct sunlight, hot engines, rain, strong wind, and low relative humidity. Core preservation materials and equipment should be close to the core handling area to facilitate a rapid operation. Accurate measurements of recovery must be made and recorded. Any recovery in excess of the core cut should be reported, as well as nonrecovery. Assign nonrecovery and overrecovery to the bottom of each core, unless some special observation indicates that an exception should be made. All such exceptions should be noted. The following data and observations may be helpful in determining the origin of overrecovery and nonrecovery:

a. Drilling parameters—drilling time, torque, rate of penetration, pump pressure, etc.
b. General conditions of the core—continuity, broken sections, induced fractures, etc.
c. Condition of the bottomhole coring assembly.

Label the core depths from top to bottom and assign overrecovery and underrecovery to the bottom. The top of the next core should be given the drilled depth. This means that in the case of overrecovery there will be the same depth in two cores. However, these cores will be distinguishable from each other because of their core numbers. Core depths must be adjusted to log depths before correlations can be made between log properties and core properties and between cored and uncored wells. Core-to-log adjustment can be made using detailed core descriptions or core scans.

It is recommended that all core barrels be laid down on the catwalk or rig floor before removal of core. The following guidelines are appropriate for laying out and marking the core:

a. The bottom of the core comes out of the barrel first and the first piece of core should be placed at the bottom of tray, box, or trough, with each succeeding piece being placed closer to the top.

b. Caution must be exercised in maintaining the proper sequence and core orientation to ensure that individual core segments are not out of place or turned upside down. Any portion of the core that is badly broken should be put in thick plastic bags and placed in its proper position.

c. Fit the core together so that the irregular ends match, then measure total recovery.

d. Do not wash the core (see 2.4, 3.5, and 3.6). If excess drilling fluid is on the core surface, it may be wiped off with a clean drilling fluid saturated cloth and wrung out as often as needed.

e. With red and black indelible markers, taped together, stripe the core from top to bottom with parallel lines (see Figure 2-1). The red line should be on the right as the individual performing the marking is facing from the bottom of the core towards the top. Arrows pointing toward the top of the core should be used to avoid confusion.

f. With an indelible marker or paint stick, starting from the top, draw a line across the core at each foot and label each line with appropriate depth.

g. To obtain reliable core analysis, speed is essential in removing, laying out, labeling, and preserving the core to minimize any alteration due to exposure (see 2.5).

h. The core should be preserved (see 2.5) and placed in numbered containers for transportation to the laboratory. It is recommended that the entire core interval be preserved at the wellsite, with sampling being reserved for the controlled conditions at the laboratory.

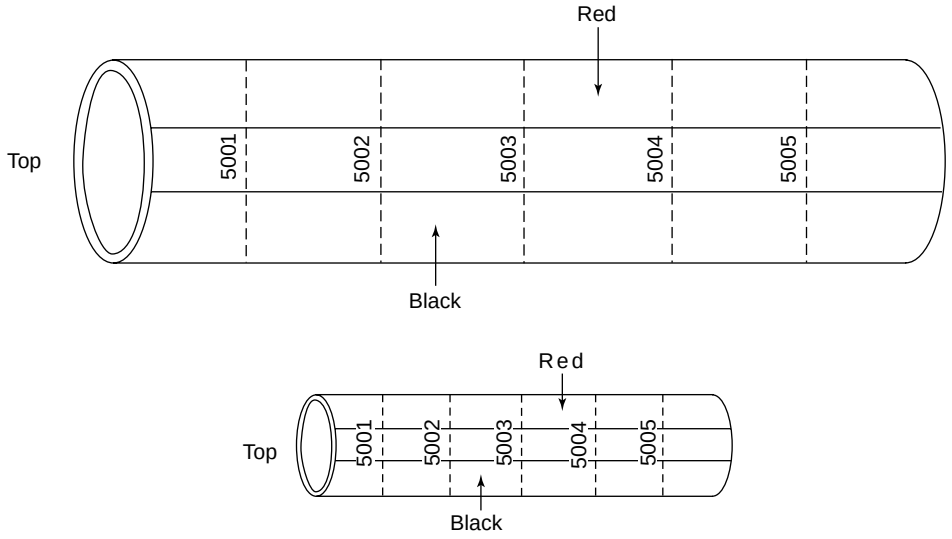

Figure 2-1—Core Marking

Even a few minutes exposure, depending on atmospheric conditions, can cause a significant loss of both water and light hydrocarbon fractions from cores. If the core is accidentally washed with water, allowed to remain in the core barrel, or let stand before preservation, then this information should be noted.

2.2.4 Handling Liners and Disposable Inner Barrels

Inner core barrel liners and disposable inner barrels improve the recovery of poorly consolidated or fractured formations. They are made of plastic, fiberglass, or aluminum and are rated to various temperatures. When coring unconsolidated or poorly consolidated formations, select the liner or disposable barrel to withstand the circulating temperature. Hard beds such as shale are best cored using either fiberglass or aluminum to prevent jamming and consequently poor core recovery. Certain coring fluid additives such as caustic react with aluminum barrels causing release of aluminum ions, that may react with the core to alter its surface properties.

When coring a poorly consolidated formation, to avoid rock compaction it is advisable to cut short lengths, 30 feet or less depending upon the rock strength. In long lengths, the lower section of core may be over compacted and damaged by the weight of the overlying material. Damaged core is of limited use for core analysis. When coring fractured formations, short core lengths may also be beneficial to decrease the risk of jamming.

The core barrel should be brought to the surface smoothly. During the last five hundred feet the core should be surfaced slowly to minimize gas expansion that can severely damage unconsolidated core if the pressure is reduced too quickly. Where gas expansion damage is expected, a perforated liner or a perforated disposable inner barrel can be used to provide a means for gas escape. All perforations must be sealed if the liner or disposable inner barrel is used as a core preservation container. Alternatively, the entire perforated section can be placed in plastic bags to prevent fluid loss.

A core-filled liner can be lowered to the catwalk, within the metal inner core barrel, using a system of pulleys anchored to the end of the catwalk. The barrel should not be banged on the rig and should be lowered gently onto the catwalk. Core-filled inner barrels flex, particularly those made of fiberglass or plastic, and should be supported by a splint. The splint should be attached to the inner barrel while it is hanging in the derrick.

a. Wedge the core barrel to prevent rotation and remove the core catcher. Transfer the core catcher material to a suitable length of liner or disposable inner barrel. The core catcher material is generally too disturbed to be used for quantitative core analysis.

b. A catwalk extension can be used to remove the entire core-filled liner from the inner barrel without flexing.

1. If the entire liner is removed or if handling a disposable inner barrel, feel inside and locate the top of the core. Cut the liner at this point. Label the core with orientation lines (red on the right and black on the left), depths, and other identification (see 2.2.3). Label depths every foot starting from the top.

2. (Preferred Method) If there is no room to remove the entire liner, pull out a little over 3 feet (0.91 meter) at a time using adequate support to prevent it from flexing. Label each 3-foot (0.91-meter) length with orientation lines and with a number to represent its position in the sequence of cut lengths. Label depths on the 3-foot (0.91-meter) lengths once the entire recovered interval is processed and the top of the core is located.

c. Cut the liners and core into 3-foot (0.91-meter) lengths using an air or electric powered circular saw. Fiberglass and aluminum liners should be cut using a mounted circular saw. Be careful to avoid vibration and rotation of the core. Clamps on the core barrel should be used with caution to avoid damaging the rock. Alternatively, the entire 30-foot (9.1-meter) length of core can be capped and shipped with a splint attached to the liner or inner barrel to prevent it from flexing. The core can then be cut in the laboratory to any specified length. This minimizes the handling of the core at the wellsite; however, preservation, sampling, and shipping procedures become more difficult.

d. Physically stabilize the 3-foot (0.91-meter) lengths using a nonreactive casting material (e.g., epoxy) to fill the annular space between the core and liner. Alternatively, the annulus can be filled with a nonreactive fluid in order to prevent evaporation. As a minimum precaution, the 3-foot (0.91-meter) lengths should be sealed with standard end caps.

e. Transfer the lengths to labeled boxes and cushion them for the trip to the laboratory. Use screws to attach wood lids to wood core boxes to avoid core damage caused by hammering.

2.2.5 Pressure-Retained Core

Pressure retaining core barrels are designed to obtain the best possible in situ fluid saturation. This method of coring offers an alternative to the conventional core barrel that loses pressure upon its retrieval to the surface. To enable the fluid saturations to be measured in the laboratory, the core must go through extensive handling. The core barrel assembly is placed in a special core service unit and the drilling fluid is flushed from the annulus between the inner and outer barrel using a nonreactive fluid while maintaining proper back pressure on the entire system. The entire core barrel assembly is then placed in a freezer box filled with dry ice (see 2.5.2.2).

To this point in the handling of a pressure-retained core, the work should be performed by trained service company personnel to their specifications.

The following procedures performed on frozen cores should be supervised by the operating company:

a. Remove the pressure-retained inner barrel from the ice, place in a safety shroud, and cut to the desired lengths.
b. Place each section back on ice, as it is cut. Take care to ensure that sections are laid down in such a way that top and bottom and position in the core section can be clearly identified. (see 2.2.3).
c. Raise one end of the core section at a time and place core cap with label on each end, securing with a hose clamp (see 2.2.4).
d. The labels should be marked with the company's name, recovered pressure, legal location, depth range of core, and processed core depth.
e. Place processed core sections in insulated shipping boxes and pack with dry ice. Mark the box with core numbers and depths along with company, location, and shipping information. If insulated shipping boxes are in transit for more than 24 hours, additional dry ice may be required.

2.2.6 Sponge Core

The sponge coring assembly is designed to improve the measurement of reservoir fluid saturations. As the core is brought to the surface, fluid that would otherwise be lost by expulsion because of pressure reduction is trapped by an absorbent polyurethane sponge surrounding the core. The coring assembly consists of 6 pre-cut 5-foot (1.52-meter) lengths of aluminum liner run within a standard steel inner barrel. The sponge core is, for the most part, then handled according to procedures established in 2.2.4. In most cases, the liner must be pumped from the coring assembly. The pre-cut liner is stored and preserved in PVC shipping tubes filled with a suitable nonreactive fluid. The PVC shipping tube is sealed with one rigid and one rubber gas expansion end cap. For orientation purposes, each sponge liner section is beveled on one end.

Once the sponge core arrives at the laboratory, it is milled open and both the core and sponge are extracted of all reservoir fluids (see 4.3.4).

2.2.7 Wireline Sidewall Coring

Wireline sidewall cores are extracted from the formation by a variety of means. Percussion sidewall coring involves the use of an explosive charge that propels a hollow projectile into the formation. Because of the forces produced by the entry of the percussion core bullet into the formation, compaction, fracturing, and rearrangement of rock grains occur; great care must be exercised when handling this core material.

Alternatively, wireline sidewall cores may be mechanically drilled from the formation with a rotary bit. Damage is minimized with this technique; however, this method is not feasible in all types of rock. If the sample breaks during removal from the coring tool, it should be pieced together and any damage should be noted. Other sidewall sampling devices involving the use of pressure-actuated coring receptacle samplers are also available.

When using sidewall coring techniques, fragile samples should be placed in plastic or glass jars with metal lids. Paper or other materials capable of absorbing liquids should not be placed in jars to act as sample cushioning material. Heat-sealable plastic laminates are an acceptable preservation technique for the drilled sidewall samples. All samples should be stabilized and cushioned during transport to the laboratory (see 2.5.2.1) and be accurately labeled (see 2.2.3).

2.2.8 Wireline-Retrieved Continuous Coring

In wireline-retrieved continuous (WRC) coring operations, the core barrel is recovered while the drill string remains in the borehole. Trip time is reduced and consequently the method can be less expensive than conventional coring. Typically, long vertical intervals are cored continuously, and certain wells may be cored from surface to total depth.

2.2.8.1 Depth Marking of WRC Cores

Good communication between the wellsite core retrieval personnel and the driller is necessary for accurate depth marking of WRC core. In conventional coring, the driller provides the top and bottom depths of the cored interval. This may cause some confusion in assigning core depths, because conventional cores are referenced from the top of the cored interval. Because WRC coring is continuous, it is best to use the bottom of the previous coring run as the top of the subsequent run. Monitoring for possible fillup between runs is suggested to minimize errors from this cause. Maintaining sound core accounting procedures in the form of a realtime spreadsheet should minimize the potential for errors in determining the top of the next interval. The spreadsheet information should include core run number, top of cored interval, bottom of cored interval, percent recovery, and a column noting the point from which core marking started.

All WRC core should be marked and labeled in accordance with 2.2.3, except assignment of depth should be modified in accordance with the following procedures. The conventional procedures for assigning core depths are not always appropriate for WRC core. In WRC coring, core dropped in one coring run may be retrieved in the next coring run, so the true depth of the current core may be in the previously cored interval. The retrieval of "dropped core" also means that the volume of overrecovered core is larger than is commonly encountered in conventional coring. Given the larger volumes of core involved, it is more important to use a core marking procedure that avoids assigning the same depth to more than one piece of core. If overrecovery occurs, the core depth marking can start with the bottom of the core, with the depth of that piece of the core being assigned the bottom reported

by the driller. Core marking should then move from bottom to top. If 100 percent or greater recovery was also obtained in the previous coring run, depth assignments should follow conventional procedures, with the depth marking beginning at the top.

For underrecovery, the following equation should be used to account for missing core:

$$\begin{aligned} \text{Missing core} &= \text{Depth Drilled} - \text{Core Length} \\ &= [CDD - PDD] - [CBL + PC - VOID] \end{aligned} \quad (1)$$

Where:

MC = missing core length, feet (meters).
CDD = current driller's depth, feet (meters).
PDD = previous driller's depth, feet (meters).
CBL = core barrel length, feet (meters).
PC = protruding core, feet (meters).
$VOID$ = void in core barrel, feet (meters).

In underrecovery, the core depths should be labeled from top to bottom, assigning underrecovery to the bottom of the cored interval.

2.2.8.2 Alternate Method for Depth Marking of WRC Cores

An alternate to the method discussed in 2.2.8.1 is to assign depths in the same manner as conventional cores (see 2.2.3). As for conventional cores, there may be underrecovery or overrecovery. Starting at the top, mark the core with depth marks every foot through to the end of the core. No attempt should be made to resolve underrecovery or overrecovery intervals before core-to-log adjustment. If depths are consistently marked from the top of each core run and each core run has a unique number, there may be the same "depth" in two consecutive cores, but they will be distinguished by their core run number. If an attempt is made to adjust for overrecovery at the wellsite, this can result in tremendous confusion with cores relabeled multiple times.

2.2.8.3 Washing of WRC Cores

As for conventional coring, care should be exercised to avoid damaging the rock; e.g., by washing it with inappropriate fluid(s). If there is any ambiguity about damaging effects, err on the side of caution, for example, by avoiding washing the core.

2.2.8.4 Analysis of WRC Cores

The extent to which the WRC core is analyzed varies from operator to operator. The core analysis procedures for WRC cores differ significantly from those used for conventional cores. In conventional coring operations, the core is returned to a laboratory for analysis and, at some future time, the core analysis results are adjusted to downhole log depths. With WRC core, much of the analysis is performed in the field, and in some cases, the core may never be archived. Typically, a wellsite geologist should describe the section with sufficient lithological detail to enable core-to-log adjustment and correlation. The geologist should note the hydrocarbon shows, porous intervals, and facies changes. Some operators use mobile laboratories through which they perform fairly sophisticated analyses of the WRC core, including core gamma scanning, ultraviolet photography, gas chromatography, and measurements of porosity, density, magnetic susceptibility, mineralogy, and acoustic properties.

2.2.9 Oriented Core Barrel

Orientation of the core barrel is accomplished using electronic, multi-shot instruments and specialized core scribing equipment. Alternatively, paleomagnetic rock signature can be used for core orientation purposes. Strict handling procedures must be followed to ensure that orientation data are positively depth correlated and matched to the proper piece of core material. This is particularly critical in fractured rock units where disposable inner barrels and liners are commonly used.

2.3 FIELD SAMPLING AND ANALYSIS

2.3.1 General

In general, sampling of recovered core material at the wellsite is not recommended. If it is necessary to sample immediately, precautions must be taken to minimize the exposure time of the core. Sampling should be quick, efficient, and performed in accordance with sound safety practices. Always obtain samples using the least damaging or contaminating method available. The entire core should be saved in all cases.

Specific reasons for wellsite sampling include, but are not limited to, a) chip sampling for lithologic description and/or mineralogical determination, b) measurement of basic rock properties, c) fluid compatibility-completion testing, d) wettability studies, e) observation of oil fluorescence/cut, f) anelastic strain recovery measurements, and g) methane desorption studies for the analysis of coal. All sections removed from the cored interval should be recorded on the field data sheet (see 2.3.3) and physically represented in continuous core sections using rigid spacers. The removed sample should be preserved, labeled, and packaged in a manner consistent with the test desired. Any additional pertinent data should accompany the samples to the laboratory or be available for wellsite analysis. Other special wellsite core analysis techniques are feasible if adequate mobile facilities are available to run tests under controlled conditions.

Hammering can damage the core and may preclude core analysis. If possible, chip samples should be taken at naturally occurring breaks in the core or with a precision trim saw. The sample size should be kept to the minimum necessary to perform the desired analysis. Place samples in individual

bags and preserve the fluid saturation with a suitable preservation technique (see 2.5).

If a detailed core examination is to be made at the wellsite, sampling and core handling should be done quickly, and only if the examination is critical to the success of the well objective.

2.3.2 Transportation and Logistics

The transport method should be expedient and provide protection against damage from environmental changes, mechanical vibration, and mishandling. Other important factors to consider when choosing the mode of transport include, a) distance and remoteness from the wellsite to the laboratory, b) onshore or offshore conditions and terrain, c) core material competence, d) weather conditions, e) type of preservation or packaging, and f) cost.

In all cases, precautions must be taken to securely stabilize the core material. In air transport, the storage cabin may not be pressurized, and this can be a factor in the core preservation. Do not stack core in such a manner as to damage the core material. Commercial carriers unaccustomed to transporting fragile materials should be used with caution. For safety reasons, cores packed in dry ice may have to be treated as a "chemical" for transportation purposes.

In all methods of transportation, a transmittal letter or contents documentation form with pertinent shipping information should accompany the shipment. A separate copy of this letter should be sent to the recipient via surface mail or fax. All applicable U. S. Department of Transportation regulations should be followed in the shipment of core materials. When standard core boxes are used they can be palletized, banded, and shipped as is. Cores, particularly those of unconsolidated materials, may be frozen or refrigerated at the wellsite for preservation and stabilization during transportation and storage. If freezing is used, the core must be entirely frozen before shipment to avoid mechanical damage. Frozen core is usually crated in insulated containers and packed with dry ice. Refrigerated core is usually shipped in self-contained refrigerator units. A temperature monitoring and recording device should accompany the core to ensure the desired conditions are maintained during transportation (see 2.5.2.2).

2.3.3 Data Sheet

A suitable data sheet should be provided for and completed by the wellsite engineer or geologist, to supply as complete a record as possible of the conditions of coring. This information will be valuable in qualifying the interpretation of the core analysis data. Further, this record may suggest either that certain additional tests be run to supplement the basic tests, or that other tests would not yield significant data. This will result in the most useful analysis for the least time and cost. Figures 2-2 and 2-3 are example forms, and the use of these or similar forms is recommended.

It is important to have as much pertinent data as possible accompany the core material. The following is a list of desirable information:

a. Well identification, API well number, elevation, vendor names and contacts, as well as telephone/telefax numbers and addresses.
b. Drilling fluid type, contents, and measured data.
c. Core type and equipment used.
d. The formation(s) cored, with the top and bottom driller's depth.
e. Designation of critical coring information and any pertinent coring notes, i.e., total coring/trip time, difficulties, and recovery.
f. Formation water salinity and production fluid data.
g. Preservation guidelines. Exposure time.
h. Analysis requested.
i. Coring log and drilling records.
j. A core description.
k. Well logs and mud logs (if available).

2.4 ROCK TYPES AND SPECIAL CONSIDERATIONS IN HANDLING

2.4.1 General

The term "rock type" is used to describe the major distinguishing feature(s) of core material. This can refer to the degree of consolidation, presence of fractures or vugs, composition (shale), or physical properties (e.g., low permeability) of the rock. Geological descriptions of rock are more complex and classification schemes have been devised to categorize specific rock types with respect to texture, type of cementation, grain size, etc. Many special considerations must be taken into account when designing a wellsite core handling program. Paragraphs 2.4.2 through 2.4.13 include general guidelines for various rock types.

2.4.2 Consolidated Rock

Consolidated rocks are hardened as a result of cementation. They need no special treatment at the wellsite. Cementation in rocks is defined as the process of precipitation of cementing materials around the solid grain surfaces. Rocks can be described as consolidated, poorly consolidated (friable), or unconsolidated, depending on the degree of compaction and cementation. Common consolidated rocks include limestone, dolomite, sandstone, and chert.

2.4.3 Unconsolidated Rock

Unconsolidated rocks have little or no cement and are essentially compacted sediments. Poorly consolidated rocks have minor cement but not enough to make them hard. These rocks are best cored using an inner core barrel liner or a disposable inner barrel (see 2.2.4). Care must be taken to prevent

Prepared By: _____ *Date:* M M D D Y Y Core Number _____

Operator _____ Coring Company_____ Core Analysis Company_____

Contact/Phone _____ Contact/Phone _____

Well Identification _____ Field/Reservoir _____ Address _____

API Number _____ ❑ Vertical Well ❑ DeviatedWell ❑ Sidetrack _____

County/State/Country _____ Legal Location/Block_____ Elevations: _____ Ground/Mean Sea Level _____ Kelly Bushing ____

Persons Requesting Work _____ Phone _____ Fax _____ Address _____

Report Results To _____ Phone _____ Fax _____ Address _____

Alternate _____ Phone _____ Fax _____ Address _____

Rig Contract _____ Phone _____ Fax _____ Address _____

Coring/Drilling Fluid
Type/Contents _____ Weight _____ ppg Funnel Viscosity _____ sec Water Loss _____ cm^3/30 min Chlorides _____ ppm pH _____ Tracer _____

Type of Core
❑ Conventional _____ Inner Barrel Type _____
❑ Sponge ❑ Oriented Barrel
❑ Pressure Retained ❑ Other _____
Sidewall: ❑ *Percussion or* ❑ *Mechanically Drilled Number Attempted* _____ *Number Recovered* _____

Analysis Planned
❑ Plug Size or ❑ Full Diameter
Method
❑ Fluid Saturation _____
❑ Porosity _____
❑ Grain Density _____
❑ Permeability _____
❑ Surface Gamma Log _____
❑ Special Instructions _____

Transportation
❑ By Service Company _____
❑ Ground _____
❑ Air _____
❑ Other _____
Carrier _____
Date shipped _____

Preservation
Method ❑ Plastic Laminate/Type _____
❑ Freeze ❑ Dry Ice or ❑ Liquid Nitrogen
❑ Refrigerate _____ °F _____°C
❑ Core Inner Barrel
❑ Core Wrap and/or ❑ Dip-Type ❑ Resination
❑ Other

Exposure Time/Climate _____
Notes _____

Laboratory/Long Term Preservation _____
Special Instructions _____
Post Core Analysis Instructions/Core Material Distribution _____

Formation(s) Depths (Drillers)
_____ Interval Cut _____
Expected Core Point _____ Interval Recovered _____
_____ Interval Cut _____
Expected Core Point _____ Interval Recovered _____
_____ Interval Cut _____
Expected Core Point _____ Interval Recovered _____

Well Inclination _____ Core Diameter _____Units_____
Total Core Recovered_____ Bit Type _____
Length Cored _____Units _____ Percent Recovery _____
Coring Time _____ Trip Time _____
Estimated Connate Water Salinity _____ ppm Chlorides or Estimated R_W _____ @ _____ ❑ °F ❑ °C
Estimated Production ❑ Oil
❑ Condensate
❑ Dry Gas

Notes _____

Attach Coring log And Core Description

Figure 2-2—Core Analysis Wellsite Data

General Information
Analysis Requested Date [M][M][D][D][Y][Y] API # _____
File # _____

Requested By: _____

❑ Full Diameter or ❑ Plug : Size _____ Units _____

Method	*Special Instructions*
❑ Saturation	_____
❑ Porosity	_____
❑ Permeability	_____
❑ Other	_____

Objective of the tests as agreed by parties involved _____

_____

Transportation and Inventory

Carrier _____ Date Shipped _____ Core Arrival Date _____

Core condition on arrival:

❑ Preserved ❑ Frozen ❑ Unpreserved ❑ Cleaned ❑ Other _____

Correlation Depths: Driller's _____ Log _____ TVD _____ Units _____

Allocation of the missing cored interval: ❑ Bottom ❑ Middle ❑ Top _____

Screening

❑ CT ❑ X-ray ❑ NMR ❑ Fluroscopy ❑ Other _____

Sample Handling

Core Gramma (yes/no, type) _____

Lab Preservation (prior to analyses) _____

Sampling Method

Sample Sleeve ❑ Yes ❑ No, Type _____

Nominal Sample Diameter/Length_____ Units _____

Plugging/Trimming Fluid_____

Treatment prior to testing (flushing, evaluation, resaluration, etc.) _____

_____

Core Disposition (removed samples, storage location, preservation, etc.)_____

_____

Lab Analyst _____

Other Service

❑ Slab ❑ Resination ❑ Photo: ❑ BW ❑ Color ❑ U.V. ❑ Other _____

Supplementary Tests _____

_____

Methods and Conditions

Cleaning:	Methods	Conditions
	❑ No Cleaning	Solvents _____
	❑ Dean-Stark	Temperature _____Units _____
	❑ Soxhlet	Pressure _____Units _____
	❑ CO2/Solvent	Time _____
	❑ Flow Through	Volume and Rate_____ Units _____
	❑ Others	Others _____
Drying:	Methods	Conditions
	❑ Convection oven	Temperature _____ Units _____
	❑ Vacuum oven	Time _____
	❑ Humidity oven	Relative Humidity % _____
	❑ Others	

Porosity: Methods

Pore Volume	Grain Volume	Bulk Volume
❑ Boyle's Law	❑ Boyle's Law	❑ Caliper
❑ Saturation	❑ Archimedes	❑ Archimedes
❑ Summation-Of-Fluids	❑ BV-GV	❑ Mercury Displacement
❑ BV-GV	❑ Other	❑ GV+PV
❑ Other		❑ Other

Conditions/Fluids

Confining Stress (Magnitude and Type)_____Units _____

Gas _____ Liquid_____

Pressure _____

Permeability:	Methods	Conditions
	❑ Steady State	Fluid Type _____
	❑ Unsteady State	Confining Stress _____Units _____
	❑ Probe	Sleeve Durometer _____
	❑ Empirical	Pore Pressure _____Units _____
	❑ Not Measured	Klinkenberg: ❑ Measured ❑ Empirical ❑ No Correction
	❑ Others	Inertial Factor: ❑ Measured ❑ Empirical ❑ No Correction
Saturation:	Methods	Conditions
	❑ Distillation Extraction (DS)	Temperature _____Units _____
	❑ High Temperature Retort	Fluids _____
	❑ Others	Water Density_____ Units _____
		Oil Density _____ Units _____
	Correction for Salt:	❑ Corrected for Water Volume
		❑ Corrected for Oil Weight ❑ Not Corrected

Quality Assurance: (Page number in report where the information is provided) _____

_____

Comments/Remarks: _____

Data Anomalies: _____

Figure 2-3—Basic Core Analysis Laboratory Data

the core from disintegrating. This includes making sure the core is brought to the surface, laid down smoothly, and preserved in such a way that it will survive transportation (see 2.5.2).

2.4.4 Unconsolidated Rock—Light Oil and Gas

It is critical to preserve unconsolidated cores containing light oil in an efficient and expedient manner. Any unnecessary movement of the core should be avoided. The two methods commonly used to preserve this rock type involve environmental methods, such as freezing or chilling and mechanical stabilization with epoxy, foam resin, etc. Unconsolidated core containing light oil is susceptible to significant fluid loss during handling at the surface. As with most rock types, by the time the core has been brought to the surface, it has undergone mechanical stress relief due to the removal of overburden pressure and has had varying amounts of gas expansion when internal pore pressure is lost. The degree to which these effects will alter the core depends on depth, reservoir pressure, oil gravity, fluid properties, sediment type, and coring procedures. Care must be taken to ensure that pressure does not build-up within the core barrel during handling. The inner core barrel liner or disposable inner barrel can be predrilled with holes [$^1/_8$ inch (3.18 millimeter) diameter] to avoid pressure buildup. Cored intervals should be limited in length to prevent possible rock damage under its own weight. As the core is raised through the upper 500 feet (152 meters) of the well, the core barrel should be retrieved at a slow rate to minimize chances of disaggregating the rock and causing damage to the core. If freezing is used to stabilize unconsolidated materials, the core should not be transported before it is fully frozen, as partial freezing can cause structural damage to the core (see 2.5.2.2).

Filling the annulus between the disposable inner barrel or liner and core with coring fluid is recommended for stabilization when epoxy or a permanent setting material is not used; however, this procedure can alter rock fluid saturation and wetting characteristics. When epoxy, resin, or foam injection are used, the drilling fluid must be fully displaced or drained from the annulus. The casting material must completely conform to and encase the core surface.

2.4.5 Unconsolidated Rock—Heavy Oil

The greatest difficulty in handling unconsolidated rocks that contain viscous heavy oil is prevention or minimization of delayed core expansion. The expansion is the result of slow gas evolution from the heavy oil, with no possibility of short-term drainage because of the low mobility to gas. Swelling of the rock may easily continue until the gas phase becomes continuous, and this may require volumetric expansion in excess of 6 percent to 8 percent. In liners, unconsolidated heavy oil sandstones will expand radially to fill empty annular space. Once the swollen core is tight in the liner, further gas evolution may cause a piston action, causing core to extrude from the ends of the liner sections leading to overrecoveries as high as 5 percent. It is tempting to simply cut off the extruded core and discard it, but this must be avoided. Extruded material is kept at the liner end from which it came, the plastic end caps may aid in keeping it slightly retained.

Improving core quality in unconsolidated heavy oil sandstones requires the following considerations:

a. Provide mechanical restraint to expansion.
b. Provide a means to allow gas drainage.
c. Provide mechanical strength to the core.

Item a involves use of liners that have an internal diameter only slightly larger [$^1/_8$ inch (3.18 millimeters)] than the core bit, to reduce radial expansion. During handling and preservation, flexing of liners, heating of core, and prolonged exposure of liner ends must be avoided. Axial restraint will help reduce the tendency to extrude the core. This restraint may be provided in a number of ways, including:

a. Rigid liner caps rather than rubber end caps, with the end caps stapled to the liner at a number of points, and secured with screw clamps.
b. Cutting liner segments to precise lengths and placing in a strong core box so that the ends of the box provide axial restraint. Alternatively, cores can be wedged into the core boxes with pieces of wood planks cut to lengths.
c. Special handling techniques devised by various operators may also be used, including special storage cylinders or axial retention methods.

Gas evolves slowly from heavy oil, and will continue for months. It is recommended that pre-perforated liners be used in all areas to shorten the gas flow path and eliminate piston effects. The pre-drilled holes [$^1/_8$ inch (3.18 millimeter) diameter] should be spaced no further apart than the radius of the liner. Post-drilling of liners is not recommended as an acceptable alternative because it prolongs core handling and may lead to damage. If required, liner and core segments may be placed in cylinders and repressured with an inert gas (N_2) to slow or stop gas evolution, and to avoid oxidation.

Freezing of unconsolidated heavy-oil core may be necessary, although in general, freezing is not well understood. Freezing has the following effects: a) it reduces gas evolution speed and volume, b) it enhances oil viscosity that restricts expansion, and c) it freezes interstitial water that gives the core some mechanical strength to restrict expansion and fracturing. Because the pore water is usually saline, the temperature will have to be reduced below –40°F (–40°C) to ensure full mechanical benefits (see 2.5.2.2).

Core handling during transportation and storage for unconsolidated materials containing heavy oil must maintain the mechanical restraint and low temperature. When preparing core sections for analysis, the temperature should be allowed to rise slowly, so that evolved gas can dissipate; mechanical

restraint should be fully maintained until the core is equilibrated, a process that may take weeks because of the high oil viscosity and low relative permeability.

2.4.6 Vuggy Carbonates

Large vugs can weaken the core material and cause difficulties with recovery. In many cases, core recovery is reduced in friable vuggy intervals. Standard consolidated core preservation methods should be used on this rock type (see 2.5).

2.4.7 Evaporites

Salt rocks are generally quite competent, and, except for their solubility, may be considered as consolidated rocks. Core containing salts in continuous sequences or as vug and fracture fillings should not be washed with fresh water under any circumstances. Because the physical properties of salt rocks can be altered by small changes in moisture content, cores containing salts must be immediately wiped to a surface dry state and preserved. Transportation and storage of cores containing salts must always be undertaken keeping the soluble nature of the material in mind. Cores of evaporite, anhydrite, gypsum, or calcite present no special core handling problems.

2.4.8 Fractured Rock

Many reservoir rocks are naturally fractured. Disposable inner barrels or liners made of aluminum or fiberglass are recommended for coring fractured rock (see 1.6 and 2.2.4). An oriented core can be useful in determining fracture strike and in situ stress direction (see 1.6 and 2.2.4).

2.4.9 Rocks Rich in Clay Minerals

Clay minerals may be present in small quantities in rocks, yet have a major impact on rock properties. Some of the major concerns in rocks containing clay minerals include:

a. The presence of smectite (a swelling clay mineral), even in very small quantities (1 percent), is of importance in core handling because of swelling potential, high cation exchange capacity, and osmotic suction potential.
b. Interstitial clay minerals can be physically mobilized by changes in fluid content, chemistry, or mechanical disturbance, leading to pore throat blocking or changes in surface wetness characteristics of pores or other physical changes.
c. Clay minerals in contact with their natural pore fluids are in thermodynamic equilibrium, and exposure to other fluids will alter this leading to changes in clay mineral activity, exchangeable cations, and consequent changes in mechanical and flow behavior.
d. Smectitic shales and sandstones may swell when confining stress is removed if free water is available, even if the free water has properties identical to the interstitial fluids.

Any excess fluid or mud cake should be immediately wiped from cores of smectitic materials and clay mineral rich materials, followed by immediate preservation (see 2.5.2).

2.4.10 Shale

In addition to the recommendations for clay mineral containing rocks (see 2.4.9), there are special issues relating to handling highly fissile shale. These materials have fissility planes of low strength that may split spontaneously, even if core is handled with great care. Once a fissile shale core has split, it may be impossible to obtain specimens large enough for core analysis.

It is recommended that fissile shale cores be handled in the following manner:

a. Avoid excessive handling or movement of the core.
b. Remove any excess water.
c. Preserve immediately to stop desiccation.
d. Masking tape or fiberglass packaging tape may be wrapped around core segments perpendicular to the fissility planes to reduce further splitting. Alternatively, heat-shrinkable plastics can be used.

Shales have low permeability and slow internal transfer of moisture will inevitably take place between beds of different mineralogy and fabric during long-term storage. If the core is totally unconstrained, this may result in delayed splitting even without desiccation or handling. Fissile shales are exceptionally sensitive to temperature changes, and should be maintained at a constant temperature during transportation and storage. Freezing of shales must not be allowed, since this leads to massive microfissuring and internal moisture movement.

Oil shales with organic chemical volumes in excess of 20 percent are sensitive to temperature and oxidation, and must be preserved with particular speed if detailed analysis is required. The much stronger matrix-supported oil shales are typically more quartzose and with organic chemical volumes less than 20 percent and in general do not require special handling.

2.4.11 Low Permeability Rock

Evaporation of fluids, a problem with all core materials, is a particular difficulty in low permeability and low porosity core where the percentage change in saturation may be much greater for the same volume of fluid evaporated. The time period before core is protected from evaporation is critical for these samples. The presence of clay minerals may make damage by evaporation irreversible in some samples (see 2.4.9).

2.4.12 Coal

In situ gas content, gas sorption behavior, permeability, relative permeability, cleat and fracture analysis, core composi-

tion, and mechanical behavior are the major interests in coal analysis for coalbed methane production. Gas desorption studies may be performed at the wellsite with desorption canisters. Procedures for handling of coal core should include instructions for these special studies. Wireline retrieved core barrels, core barrels with disposable inner barrels or liners, and pressure-retained core barrels have been used to cut coal cores.

Gas content and gas desorption rate are commonly measured from coal by canister desorption methods using conventional cores, mechanically-drilled sidewall cores, wireline-retrieved continuous cores, or drill cuttings. Coal segments are sealed in a canister and isothermally maintained while the volume of gas evolved from the sample is measured. Gas content measurement by canister desorption requires an estimate of gas volume lost while bringing the core to the surface and before sealing the samples in the canister. Gas content must be normalized to a mineral free matter basis. Since the gas evolved will not be 100 percent methane, the composition of the gas must be analyzed.

Pressure-retained core technology, that does not require an estimate of lost gas, offers a more accurate means to determine the total in situ gas content of a coalbed. The volume of gas evolved from the coal core in the pressure barrel is measured as a function of time, temperature, and pressure. If pressure-retained methods for coring coal are used, a special internal temperature sensor is incorporated, and no coring fluid flushing is carried out (see 2.2.5). In one procedure, the entire pressure-retained barrel is returned to bottomhole temperature and allowed to equilibrate for up to several days before the pressure is allowed to dissipate and the device is disassembled. During the temperature and pressure dissipation phase, changes should be gradual to minimize both the magnitude of the pressure gradient and the temperature gradient. The former may cause the coal to fracture internally, whereas rapid cooling may cause tensile cracks to open in the outer part of the coal core. These may affect both permeability and mechanical behavior. Alternatively, the pressure-retained coal core can be cut into one foot sections and placed in desorption canisters for measurement of the gas content and gas desorption rate.

Coal core must be handled with care at the wellsite because of its heterogeneous nature, and because the methane found in small pores within the core is under pressure. Careless treatment, liner flexing, or a sharp blow to the core barrel may cause the core to fragment, rendering the core of little use for core analysis. The internal gas pressure may aid this tendency to deteriorate, and time is required to permit gas dissipation. Coring fluids in contact with coal intended for fluid flow or mechanical tests should not contain materials capable of altering the coal structure.

Fresh coal oxidizes when exposed to air, potentially changing its surface properties and sorption characteristics. Exposure time to air must be minimized, and special handling may include canisters to receive coal core segments, followed by flushing of the cylinders with inert gas or methane.

White or yellow pens or paint crayons should be used to mark the surface of the core lengths. As with any core, preservation of moisture and minimization of exposure time is advised. Freezing of coal core is not recommended, nor is it considered necessary.

2.4.13 Diatomite

Diatomites are generally high-porosity, low-permeability rocks composed of opaline-quartz phases with varying amounts of detrital material. Diatomites are cored with disposable inner barrels or liners (see 2.2.4).

Diatomite can be preserved by environmental means, wrapping, etc. Freezing of diatomite is not recommended. Temperature should be controlled to maintain a constant temperature [35 to 40°F (1.67 to 4.44°C)] during wellsite and transportation operations.

2.5 PRESERVATION OF CORES FOR ANALYSIS

2.5.1 General

The preservation of a core is an attempt to maintain it, prior to analysis, in the same condition as existed upon its removal from the core barrel. In the process of cutting a core, recovering it, and bringing it to the surface, the fluid content of the rock is altered by unavoidable changes in pressure, temperature, etc. Pressure-retained core methods attempt to minimize these effects (see 2.2.5). Careless or incorrect practices in handling and preservation cause further alteration of the core and its fluids, thereby making the core even less representative of the formation.

Preservation and packaging of cores may vary depending upon the test(s) required, the length of time before testing, and the potential of performing wellsite tests. If the core samples are to be used to determine fluid saturation or for special core analysis, it is necessary that they be preserved for transportation to the laboratory. Evaporation and migration of fluids as well as oxidation within the sample must be avoided to obtain reliable core analysis. An additional objective of the preservation program is to prevent breakage of the cores during shipment and storage. Consolidated core may be durable enough not to require special handling procedures. However, special care should be taken with unconsolidated or fractured rock, etc. (see 2.4).

The use of unprotected glass jars, easily deformable plastics (if not properly stabilized), paper cartons, non-rigid containers, and air-tight cans are not recommended for core preservation purposes.

2.5.2 Methods of Preserving Cores

There is no one best preservation method. Experience can help determine the most satisfactory method for the rock type

in question. The choice of method will depend on the composition, degree of consolidation, and distinguishing features of the rock. Therefore, general use of one specific method of preservation will not apply to all rock types. The techniques required to preserve cores for testing may depend upon the length of time for transportation, storage, and the nature of the test to be performed. Some variation in the method of preservation may depend upon whether the cores will be analyzed locally or whether they must be prepared for long-distance shipping. Preferred methods to preserve cores for laboratory analysis include one or more of the following:

a. Mechanical stabilization.
b. Environmentally controlled preservation using chilling, regulated humidity, or freezing, if necessary (see 2.5.2.2).
c. Heat-sealable plastic laminates.
d. Plastic bags.
e. Dips and coatings.
f. Sealing in disposable inner barrels, liners, and tubes.
g. Anaerobic jars.

2.5.2.1 Mechanical Stabilization

All rock types should be mechanically stabilized prior to shipment to the laboratory. This is particularly true for unconsolidated rock (see 2.4.4 and 2.4.5). Core that has been cut using plastic, fiberglass, or aluminum liners/disposable inner barrels can be cast using resin, wax, or foam to fill the annular space between the core and the sleeve. Resin has low viscosity and will fill fine fractures. However, it is only poured into the annulus and is not under enough pressure to displace pore fluids in the rock and therefore does not impregnate the core.

Mechanical stabilization for well consolidated cores may also be as simple as wrapping the core in bubble wrap or other suitable cushioning materials. All core material should be considered fragile and handled carefully (see 2.3.2). Care should be taken to avoid disturbing poorly consolidated or fractured core prior to mechanical stabilization.

2.5.2.2 Environmental Preservation

Controlling environmental conditions to which the core is subjected by chilling or maintaining a humid environment can help to preserve the core (refer to core preparation information in Section 3). Core chilling is used primarily to minimize fluid evaporation and provide mechanical stabilization. This technique is useful in preventing the core from drying; however, its effectiveness is subject to the coring fluid type and the reservoir rock and fluid properties. When chilling core, it is still necessary to mechanically stabilize the rock for transport to the laboratory (see 2.5.2.1).

Cores that are preserved by freezing should be frozen by application of dry ice, liquid nitrogen, or placement in an electrically-operated freezer unit. Freezing may result in the migration or diffusion of fluids within the core structure or breakage of the core. Freezing can cause significant evaporative losses through sublimation. Unconsolidated cores that have been frozen can be packed with a $^1/_4$ inch (6.35 mm) thick layer of frozen brine (surface ice cake) to reduce the sublimation process. This measure is critical if freezing is used for long-term core storage. Structural damage to the core may occur if it dehydrates while frozen.

The practice of freezing core is most common for unconsolidated rocks (see 2.4.4 and 2.4.5). The full effect of freezing on a core's petrophysical properties is unknown. The freezing of consolidated core with interstitial water is not well understood. Expansion of ice crystals may cause irreversible structural damage to core. Freezing may affect the properties of rock flushed with fresh water more than those flushed with saline drilling fluid filtrate. These effects will decrease with decreasing water saturation. If it is necessary to allow the core to warm to ambient temperature before testing, the condensation of moisture from the atmosphere onto the core surface must be prevented. Thawing of the core may cause some redistribution of the fluids within the core matrix.

The fluid saturation and reservoir (mineral) properties can also be preserved by controlling the relative humidity of the core environment with specially designed ovens. This technique has wide applicability and is most effective with rocks containing moisture sensitive clay minerals and/or chemically bound water contained in minerals (see 2.4.9).

2.5.2.3 Heat-Sealable Plastic Laminates

Several heat-sealable plastic laminates are available. Aluminum foil or mylar may be used to add rigidity to the laminate. The laminated core preservation package should act as an impermeable barrier to water vapor and gases, and be resistant to chemical alteration and degradation by fluids. Laminates are easy to use and the preservation process can be performed quickly. Care should be taken to prevent tears or punctures in the laminate. A clean, flat surface is required for smoothing the laminate prior to sealing. All core should be prewrapped and taped with durable plastic or other material to cover the core ends and sharp edges. The packaged core segment should be labeled with well and depth information.

The heat sealing process is critical to the success of using this preservation method. The heat sealer must be set to the proper temperature in accordance with manufacturer's specifications to obtain an effective seal. Any discontinuity in the seal will negate the barrier properties of the material. Some laminates are available in tubular form that requires the sealing of only two ends rather than four. The head space in the preservation package should be minimized; however, enough material should be used to prevent weakening if the package will be opened and resealed more than once. In some cases, it may be advisable to evacuate the gas space where the loss of light-end hydrocarbons is not an issue. An inert gas such as nitrogen can also be blanketed over the rock to minimize oxi-

dation. When core material degasses, the laminate package will inflate; this will pose no problem if the package is sealed properly. If necessary, the evolved gas may be sampled directly with a standard gas syringe and the package resealed. The preservation package must be labeled and mechanically stabilized for shipping (see 2.5.2.1). At no time should the preservation package be subjected to extremes of temperature.

2.5.2.4 Plastic Bags

Plastic bags are only recommended for short-term preservation. Core samples should have a minimum of air space between the core and bag wall. Any excess bag can be folded against the core wall and taped to assure a tight fit. As always, clear labeling and proper stabilization procedures should be followed.

2.5.2.5 Dips and Coatings

Dips and coatings are used when cores are not to be tested within a few hours or days and when the material is to be transported over long distances. Dip coatings can also be used with plastic laminates to add mechanical integrity.

CAUTION: Cores should never be dipped directly into any molten wax or plastic material.

All core should be prewrapped with a heat-sealable laminate or plastic film and aluminum foil prior to dipping. All core segments should be labeled with well and depth information. The purpose of the plastic film wrap is to prevent contact of the core and pore fluids with the aluminum foil outerwrap. Such contact can cause oxidation of the foil and loss of its moisture and oxygen barrier properties. The following procedures should be used with the wrap and dip method:

a. Prepare a heating vat for dip-coating several hours prior to preserving the core. Observe all safety precautions. Follow the dip manufacturer's recommendations for handling. Overheating the dip can cause the coating to be ineffective.

b. Wrap the core tightly in plastic film that will conform to the surface of the rock, crimping the free ends together. Several layers of high quality plastic film are desirable to prevent puncturing.

c. Wrap the core with several layers of aluminum foil, crimping the free ends together. Avoid puncturing the aluminum wrap.

d. Tie a wire around the core to make a handle.

e. Dip the foil-wrapped core sample in the molten coating material. A liberal amount of dip coating should encase the core; a $^1/_8$- to $^1/_4$-inch (3.18- to 6.35-millimeter) thick coating is recommended. This is accomplished through the use of multiple dips, allowing each dip coating to harden prior to the application of additional dip material. It is recommended that the dip coating be hardened by suspending the core in air by means of the wire handle.

f. The wire handle should be cut flush with the dip coating. Additional dip should be applied to the wire end to eliminate a pathway for evaporation or oxidation.

Coating material must have certain properties, as follows:

a. It must be dimensionally stable over long periods of time.

b. It must not react with oil or water and not contain acids, oils, solvents or any other liquid that may be exuded when set.

c. Permeability to gases, oils, and water must be low when set.

d. It should have a low melting point, preferably below 200°F (93.3°C) maximum and have a fairly low viscosity when melted. Higher melting points are acceptable if exposure time to the dip is minimized.

e. When removed from heat and exposed to ambient conditions, it should be dry and set tack-free within 5 to 15 seconds.

f. When set, it should be tough but pliable, slightly elastic but with good tensile strength, and not melt at temperatures below 180°F (82.2°C).

As with all core preservation methods currently used, the long term effectiveness of dips and coatings remains uncertain.

2.5.2.6 Disposable Inner Barrels, Liners, and Rigid Tubes

A convenient means of core preservation is possible when disposable inner barrels or liners made of plastic, aluminum, or fiberglass are used (see 2.2.4). The core may be preserved as-is by sealing the ends of the cut inner barrel or liner. This is not recommended as a long term preservation method, but will allow the core to be processed quickly and without special equipment. Holes in the inner barrel should be sealed prior to shipping. The core can be rapidly shipped to the laboratory for sampling and testing, and, if required, additional preservation of the sealed containers is possible (see 2.5.2.2). When a conventional core barrel is used, steel, aluminum, or plastic tubes with suitable couplings, caps, or O-ring seals may be used to preserve the core. To be used effectively, the tube and end pieces must be nonreactive with the core and its fluids. Segments may be stabilized according to guidelines established in 2.5.2.1 to minimize damage in shipment.

2.5.2.7 Anaerobic Jar

Immersion of the core in liquid within an anaerobic jar can be used to prevent oxidation, evaporation, and drying during the handling of core. The anaerobic vessel is an elongated jar with a sealable lid, into which a liquid can be introduced and any free oxygen removed. The immersion liquid must be compatible with the core and pore fluids, and be able to main-

tain the current wettability of the sample (see 2.6). Typically, the following fluids are used for immersion:

a. Deoxygenated formation brine or synthetic formation brine with biocide.
b. Crude oil.
c. Depolarized refined mineral oil.

As always, follow all safety precautions when using anaerobic jars for preserving reservoir samples.

2.6 RECOMMENDATIONS FOR CORE HANDLING TO PRESERVE WETTABILITY

2.6.1 General

Wettability alteration can take place during coring, core treatment at the wellsite, or during the period of storage before measurements are made in the laboratory. Only core treatment at the wellsite is discussed here, although the measures recommended can only be successful in preserving wettability if adequate precautions are taken at each step of the core recovery and analysis process.

The validity of many laboratory core tests depends on maintenance or reestablishment of reservoir wetting conditions; however, there is rarely an initial reference point to define in situ wettability. The practices recommended here are intended to provide measures of wettability as early as possible after removal of core from the formation so that a reference point for wettability is established. Cores should not be exposed to air for any longer than is necessary.

Wettability tests should be made at the wellsite. A simple observation of imbibition of water and oil droplets placed on the core surface should be recorded routinely. In many cases, this will be the extent of wellsite wettability testing, but much more comprehensive tests can be initiated at the wellsite as well. Some special facilities would be required, including:

a. The capability to cut core plugs from newly recovered whole core. Any delay in cutting core plugs allows capillarity and diffusion to distribute surface active drilling fluid constituents to the uninvaded portions of the core. Synthetic reservoir brine or refined laboratory oil should be used as the cutting fluid.
b. Core holders and core flooding equipment to allow flushing of plugs are needed. It may be possible to avoid damage done by an unstable oil phase (one from which asphaltenes or paraffins are deposited because of changes in pressure and temperature) if the oil phase is quickly replaced by a stable, non-damaging oil.
c. Imbibition test facilities are also needed since tests of imbibition rate and extent are the best indicators of wettability in porous media.

Requirements for maintaining wettability will vary from one reservoir to another and to some extent will have to be experimentally determined. Suitable core preservation methods to control fluid loss are described in 2.5.2.

2.7 PRECAUTIONS

2.7.1 General

The fundamental objective of core analysis is to obtain data representative of in situ reservoir rock properties. Coring, handling, and preservation should be conducted in such a manner as to prevent both loss of the interstitial fluids and contamination with foreign fluids. The core should never be washed with water or oils prior to preservation. Suitable coring and handling procedures must be adopted to obtain laboratory data that are meaningful. Alteration of the rock can occur during coring, handling, preservation, sampling, and preparation prior to or during analysis. For a reliable determination of the fluid content of cores, a uniform procedure must be designed for handling and preservation. It should be emphasized that a properly designed program will benefit not only the near term user, but also future users of the core material. Some precautions in the handling and packaging of samples are listed:

a. All cores should be preserved as soon as possible after removal from the core barrel. Even after preservation, the samples should not be exposed to extreme conditions. The long term effectiveness of core preservation materials currently in use is unknown. A tested and trusted material should be used if the core is to be stored for extended periods of time.
b. Always minimize the gas head space in core preservation containers to prevent evaporation losses during core storage. This procedure will also minimize condensation losses on the inside surface of the container and help prevent breakage of the more loosely consolidated samples during shipment.
c. To minimize fluid loss, do not contact the core with cloth, paper, or any other dry material with fine capillaries.
d. Do not dip or coat the core directly with any fluid.
e. Follow stringent handling instructions for the processing and preservation of unconsolidated core (see 2.2.4 and 2.5).
f. Do not preserve an unconsolidated rock or other rock type in the same container as a rock of a vastly different lithology. This will minimize the potential of mechanical damage to weak samples.
g. Should any core be exposed to harsh handling conditions or washing, this should be noted. All pertinent data must be supplied with the core (see Figures 2-2 and 2-3).
h. Label each preservation container properly. In cases where confidentiality is an issue, this data should be coded with numbers and referenced to a master list.
i. It is recommended to have a company representative familiar with wellsite handling and preservation present during the coring operation. If this is not possible, explicit written instructions should be given to the service company

representative responsible for this activity. Upon arrival at the laboratory, cores should remain in a preserved state until ready for analysis.

j. ALL APPLICABLE SAFETY REGULATIONS MUST BE FOLLOWED WHEN HANDLING CORING EQUIPMENT AND CORE MATERIAL. The wellsite team should be protected, as necessary, against exposure to hazardous material using overalls, gloves, protective eyewear, etc. A hard hat, steel-toed shoes, and hearing protection are also recommended. Where toxic gases such as hydrogen sulfide are present, appropriate personal protective breathing apparatus must be available. Safety training is required for all wellsite workers prior to handling coring equipment, processing equipment, machinery, and core material.

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SECTION 3—CORE SCREENING AND CORE PREPARATION

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3 Core Screening and Core Preparation

3.1 GENERAL

The recommendations included in this document may involve the use of hazardous materials, operations, and equipment. This document does not address all of the relevant safety problems associated with its use. It is the responsibility of the user to establish appropriate safety and health practices prior to use and to comply with all applicable regulatory requirements regarding use and disposal of materials and equipment.

3.2 CORE DESCRIPTION

3.2.1 Principle

The purpose of core examination and description is the recognition of lithological, depositional, structural, and diagenetic features of whole or slabbed core. Qualitative and quantitative core descriptions provide the basis for routine core analysis sampling, facies analysis, and further reservoir studies such as reservoir quality and supplementary core analyses. A description provides a permanent, accessible record of the core.

3.2.2 Apparatus and Supplies

The following equipment is recommended for use in standard descriptions of the core:

a. Logging form for systematic data collection.
b. Microscope or hand lens.
c. Length measurement scale.
d. Grain size scale.
e. Appropriate chemicals such as:
   1. Water or brine to improve visibility of geological structures.
   2. Dilute HCl acid to identify carbonate minerals.
   3. Alizarin red to differentiate calcite and dolomite.
   4. Hydrocarbon solvents to facilitate the detection of oil fluorescence under ultraviolet light.
f. Coring log, drilling report, mud logs, well site information on lost core.
g. Core gamma log.
h. Ultraviolet light.

3.2.3 Precautions

The following precautions should be observed:

a. If the samples being described have been preserved for special testing, exposure of the samples to air and chemicals should be avoided until tests on the preserved core have been completed.
b. Choose an appropriate logging format. The objective of a logging form should be to accurately represent the core. When describing core, it is important to collect and record data in a systematic manner.

3.2.4 Procedures

The following procedures should be used:

a. Lay out the core to be analyzed on a viewing table.
b. Check the amount of core with the coring report and ensure that no core material has been lost during transportation. Note any damage or alteration to the core during well-site handling and transportation.
c. Check the numbering and order of boxes or containers against cumulative depth.
d. Check core continuity and orientation with respect to the top of the core. If the core has been marked in accordance with 2.2.3, the top of the core is up when the red scribe mark is on the right.
e. Check the order of core segments in the boxes; look for matching “breaks” or scribe marks from one piece to the next and from one box to the next.
f. Measure and mark footage on each box. Mark core to the nearest $^1/_2$ inch (1 centimeter).
g. If downhole gamma-ray logs are available, a comparison should be made with the core gamma-ray logs (see 3.3) to verify that the cored interval depths are consistent with well log depths.
h. Assess the entire cored sequence before commencing. Seek out distinguishing features such as units, contacts, unique markers (e.g., bentonite, coal).
i. Record major features such as the following using standardized nomenclature and abbreviations (see Section 8):
   1. Core lithology (shale, sandstone, limestone, etc.).
   2. Color.
   3. Bedding (thickness, bed contacts, erosional markers).
   4. Obvious sedimentary structures.
   5. Texture (grain size, angularity/roundness and sorting).
   6. Composition (grains, cement, fossils).
   7. Porosity types.
   8. Diagenetic and tectonic features.
   9. Note any oil stain and related fluorescence.
j. Minimize centimeter-by-centimeter descriptions. Some fine scale features may be important and should be recorded.
k. Record nonreservoir rocks, e.g., mudrocks, in addition to potential pay sequences. Subtle changes in lithology may assist correlation between core and well logs and definition of relevant zonation.

l. Record fracture information such as width, extent, density, fracture orientation if core is oriented, and presence of cementation or mud. Where possible, differentiate between natural and coring induced fractures.

3.3 CORE GAMMA LOGS AND CORE SPECTRAL GAMMA LOGS

3.3.1 Principle

Naturally occurring gamma-ray emitters (radiogenic daughters of uranium and thorium along with potassium-40) give a measurable gamma-ray response that can be recorded with depth. If such a record measured at the surface is compared with the gamma-ray readings taken from a downhole gamma-ray log, the results can frequently be used to adjust the depth of the core to coincide with the open-hole logging depths and to identify zones where core has been lost.

3.3.2 Apparatus

The recommended apparatus consists of a conveyer for moving core, lead shielding to reduce background gamma radiation, and suitable gamma-ray detectors. A typical detector system consists of a shielded scintillation crystal coupled with a photomultiplier. The scintillation crystal is normally sodium iodide doped with thallium [NaI(Tl)]. Other scintillation crystals include cesium iodide (CsI) and bismuth germanate (BiGeO).

Signals from the detectors are processed and the gamma-ray events are energy-sorted and counted. Standard gamma-ray units should provide total gamma counts in APIU units. With the spectral gamma units these counts are converted into potassium, uranium, and thorium concentrations, and standard gamma-ray log API units. Signals are displayed on a computer graphics screen or plotted in downhole log formats and scale for direct comparison with downhole logs.

3.3.3 Procedures

The following procedures should be used:

a. Set the recorder readout to correspond to the scale used with the downhole gamma-ray log.
b. Log the core gamma-ray response from bottom to top. This conforms with the sequence used in downhole logs.
c. Core gamma-ray logging should be carried out smoothly, without interruption, in the correct order and spacing with matching core breaks fitted together.
d. Record a repeat section near the bottom of the core to establish the reproducibility of the measurement.

3.3.4 Advantages

Core gamma-ray logging is widely available and is used in general practice to correlate core depth with log depth. The spectral gamma-ray apparatus will differentiate uranium, thorium, and potassium concentrations and can be used to better identify and differentiate shale, particularly in sandstone cores with large amounts of potassium feldspar and mica.

3.3.5 Limitations

This technique is unable to detect low gamma-ray activity and can suffer from significant background interference. Analysis of samples for spectral gamma-ray response requires a slower rate of logging than that used to determine total gamma-ray response.

3.3.6 Calibration

The apparatus should be calibrated before evaluating any core material. Calibrations are sensitive to the size of core and the gamma-ray energy range. Calibration should be done by measuring the gamma-ray response to calibration tubes containing pure samples of the elements of interest. The total gamma-ray apparatus requires a calibration tube containing potassium (K-40), uranium (U-238), and thorium (T-232) of known activities. The calibration procedure for the spectral gamma unit requires the same elements but in individual tubes. A blank calibration tube should be measured to ensure a minimal amount of background gamma-rays are interfering with the apparatus. The speed of the conveyor should be slow enough, to provide an acceptable ratio of signal to background noise. A ratio of three times the square root of the count rate to background count rate greater than one is acceptable, i.e.:

$$\frac{3 + \sqrt{\text{count rate}}}{\text{background count rate}} > 1$$

3.3.7 Accuracy

The precision of the measurement varies with the square root of the count rate.

3.4 CORE IMAGING

A recorded image of the core is essential. This record will provide information that can be used if viewing of the core is not possible. The record may include visual images of the surface features of the core using photographic techniques, visual representations of the internal structures of the core such as x-radiographs, computed x-ray tomographs, magnetic resonance images, or acoustic images. Any record should include information on core depth along with a scale or legend indicating intensity range of the recorded image.

In addition to photographic representations, modern imaging techniques provide quantifiable information for the core that can be used as the basis for routine core analysis sampling, facies analysis, reservoir quality studies, supplementary core analyses, and downhole log interpretations.

3.4.1 Photography

3.4.1.1 Principle

Core is normally photographed under both natural (5,500 K) and ultraviolet light (254-365 nm) along with a standard color scale. The natural light photographs show lithology and sedimentary structures and allow specific features recorded in the core description to be examined. Ultraviolet light photography can highlight hydrocarbon-bearing zones by causing most oils to fluoresce in shades ranging from orange-brown for heavy oils to bright yellow for high gravity oils; condensates can appear as a very light white to blue-white color. Non-hydrocarbon bearing zones will appear as purple regions, although some minerals such as chalky-limestones also appear purple.

3.4.1.2 Advantages

Core photography provides a visual record of the core that can be used to reconstruct damaged core pieces, minimize core handling, and identify the location of samples if photographed following sampling.

3.4.1.3 Limitations

Photographic colors may not represent true colors of the core. Highlighting of features may require wetting the core surface for photography. A scale and color bar are required if adjustments are necessary.

3.4.2 X-Ray Techniques

3.4.2.1 Principle

X-ray techniques can be used to noninvasively examine the internal character of a core. An x-ray beam is directed through the core and variations in attenuation of the incident x-ray beam are measured. In order of increasing definition, these techniques are: fluoroscopy, x-radiography, and computed tomography (CT scanning).

The utility of these methods relies on their sensitivity to density contrasts within the core. Consequently, non-porous, dense areas will be contrasted against porous, low-density areas. High definition of density variations within the core requires multi-directional measurements or spatial separation of the contrasted areas.

3.4.2.2 Fluoroscopy

In this technique, the core is moved across an x-ray source. The beam, attenuated by the core, impinges on a fluorescent screen, is intensified, and is recorded by a video camera. The captured image can be viewed on a monitor, recorded on a video cassette, and converted to digital format for later processing and viewing. The output from this technique is a continuous image along the length of the core.

Because the core is moving, the resolution of the resulting image may be diminished. It does, however, enable identification of density variations, some geological structures, and areas of lost core within plastic or fiberglass sleeves. It is also useful in identifying fractured and badly broken zones.

3.4.2.3 X-Radiography

A core sample is positioned between an x-ray source and an x-ray sensitive film. Attenuation of the x-ray beam by the core is captured on film. The developed film can be viewed on a light table, or used to make a black and white print.

It is possible to obtain multiple images of the same piece of core and display them side-by-side. The core should be first x-rayed to obtain an initial image and is then rotated 90 degrees in order to obtain a second image. This is useful in orienting sleeved core prior to slabbing because it can establish the bedding direction.

During x-radiography, the core sample is stationary and provides a higher resolution image than the fluoroscopy method. X-radiography enables detection and evaluation of internal geological structures such as bedding planes, fractures and nodules, lithological changes, and bulk density.

3.4.2.4 Computed Tomography (CT)

In CT, the core is scanned by a highly collimated x-ray beam. Detectors on the opposite side of the core measure the intensity of the transmitted beam. The x-ray source and/or detectors rotate or translate around the sample. A series of x-ray attenuation measurements are made and numerically reconstructed to give the spatial distribution of x-ray attenuation coefficients within the sample.

The resolution of the image depends on the thickness of the x-ray beam, the number of detectors in the scanner, and the size of the pixel array used to reconstruct the image. X-ray beam thickness varies from about 2 millimeters to 10 millimeters. CT enables detection and evaluation of internal geological structures such as bedding planes, fractures and nodules, lithological changes, and bulk density. Combination of sequential x-y scans along the core axis can provide slab images or three-dimensional images of the core.

3.4.2.5 Advantages

X-ray techniques provide quantified, objective representations of the core. These representations can provide similar advantages as described for core photographs in 3.4.1.2 without the need for exposing a surface of the core.

3.4.2.6 Limitations

Resolution of the images is less than that provided by photographs. X-ray attenuation may vary with mineralogy, depending on the energy of the x-ray beam. Some applications are still in the development stage. Further development

of the relationships between rocks and x-ray attenuation may extend these methods to other areas of core analysis.

3.4.3 Nuclear Magnetic Resonance (NMR)

3.4.3.1 Principle

NMR imaging is used to provide a reconstructed image of fluids within a core sample. NMR measurements are based on the observation that when the proper radio frequency excitation energy is applied to an ensemble of nuclei, a portion of nuclei at a lower energy level can be promoted to a higher energy level. When the excitation is removed, the rate at which excited nuclei return to the lower level can be detected and measured. The excitation energy is supplied by an oscillating magnetic field in resonance with the nuclei. The nuclei response is measured through a tuned receiver coil. The location of the excited nuclei can be determined by the application of magnetic field gradients during the excitation and detection processes.

The primary nucleus used in core analysis applications is ^{1}H. Other nuclei that can be imaged include, but are not limited to, ^{2}H, ^{31}P, ^{23}Na, ^{13}C.

3.4.3.2 Advantages

Magnetic resonance images are noninvasive and provide an image showing fluid locations within a sample. The advantages described in 3.4.2.5 apply provided the sample is contained within a suitable non-metallic holder.

3.4.3.3 Limitations

This technique is not descriptive of the core but of the liquids within the core. The technique requires a high density of resonant nuclei for an adequate signal. Hence, low porosity cores will give weak signals. Parmagnetic or ferromagnetic minerals may inhibit or degrade the measured signal and can distort the image.

3.5 CORE SAMPLING AND CORE PREPARATION (BASIC CORE ANALYSIS)

3.5.1 Principle

The sampling procedure for basic core analysis is determined by the type of information required. Sampling will generally take into account one or more of the following:

a. Lithological distribution.

b. Porosity and permeability variations within the lithological units.

c. Distribution of hydrocarbons.

3.5.1.1 Plug Samples

Referred to as plugs, should be removed from sections of whole core oriented either vertically or horizontally with respect to the whole core axis or with respect to the normal of the bedding planes. These plugs will provide data on matrix properties. See 3.5.2.4 for procedures for removal of core plugs.

3.5.1.2 Full Diameter Samples

Full diameter samples (sections of whole core), in addition to plug samples, should be taken in the following types of zones or where there are significant large scale heterogeneities that are different from the matrix properties. These can occur in, but are not limited to:

a. Vuggy carbonates—Vugs can bias the measurement of porosity within a sample. Samples should be taken to obtain a representation of the relative proportion of vugs-to-rock in the reservoir.
b. Fractured reservoirs—Fractured zones may be difficult to sample due to the fragile nature of the rock. However, natural fractures can have a significant positive impact on reservoir permeability and should be sampled where possible or appropriate.
c. Conglomerates—Within a conglomerate, if the pebble size is large with respect to the sample size, sample size can greatly bias rock property measurements. To obtain a representative sample of a unit of conglomerate it is necessary that sample size be sufficient to include all pebble sizes.

3.5.2 Sample Cutting, Trimming, and Mounting

3.5.2.1 Principle

The core should be cut and trimmed to provide regularly shaped samples, most commonly right cylinders. Unconsolidated, disintegrating, and very friable samples may have to be mounted prior to testing. Regardless of the degree of lithification, sample handling should be kept to a minimum.

3.5.2.2 Apparatus and Supplies

The following equipment and supplies are commonly used in core sample cutting and trimming operations:

a. Large slab saw with a diamond blade.
b. Trim saw with a diamond blade.
c. Drill press with diamond-edged core drills, capable of drilling cylindrical samples.
d. Dressing wheel or saw for squaring plug ends.
e. Fluid pumps for delivering various coolants (brine, oil, air, water, liquid N_2) to the cutting surfaces.
f. Indelible marking medium such as India ink.
g. Sleeves of lead, aluminum, or heat shrinkable plastic for mounting soft, unconsolidated, or very fragile samples.

The following information should be available prior to cutting, trimming, or mounting operations:

a. The total number of samples required.

b. Size and orientation needed.

c. Exact depth locations for samples and how they are to be labeled.

d. Fluids to use for cutting samples (cores containing clays or shale laminations sensitive to fresh water will deteriorate unless proper drilling fluids are used).

e. Available core images.

f. If required, materials for the preferred method of preservation for both plugs and core from which the plugs are cut.

3.5.2.3 Precautions

The following precautions should be enforced during sample cutting, trimming, and mounting operations:

a. Saw and drill press operators should wear protective earplugs, goggles, gloves, and other necessary safety equipment.

b. Lubricant/coolant flow should be sufficient to cool the drill bit or saw blade and remove the cuttings without eroding the sample.

3.5.2.4 Procedures

The following procedures for preparing various type core samples should be followed:

a. Plug samples:

1. Drill plugs at specified points using the appropriate size bit. Care should be taken to drill straight plugs. If too much pressure is applied during the drilling operation, the bit will flex causing the plug to deform.

2. Trim plugs to the required length making sure that the ends are parallel. Keep and label trimmed ends.

3. Label, preserve, and/or store samples, as required.

b. Full diameter samples:

1. Cut sections of the core selected for analysis slightly longer than required to allow for finish grinding operations.

2. Remove barbs and smooth chipped edges by dressing the ends. Cylinder ends should be as close to parallel as possible.

3. Clearly mark the samples and preserve or store as required.

c. Unconsolidated samples—There are two distinct methods of preparing unconsolidated cores. The procedures depend on whether the core is stabilized by freezing, epoxy impregnation, or both or is unstabilized. Stabilized cores are generally drilled while unstabilized cores are plunge cut.

1. Stabilized cores—Frozen cores are obtained using the practices listed for plug samples, with appropriate modifications to avoid thawing of the core:

(a) Pre-cool the core with dry ice and drill with liquid N_2 coolant.

(b) Drill plugs at specified points using the appropriate size bit. Care should be taken to drill straight plugs. If too much pressure is applied during the drilling operation, the bit will flex causing the plug to deform. Ensure that there is adequate liquid nitrogen flow so that the sample is kept frozen and the cuttings are being removed.

(c) Trim plugs to the required length using a liquid N_2 cooled saw blade, making sure that the ends are parallel. Keep and label trimmed ends.

(d) Mount plugs in a suitable pre-weighed sleeve with pre-weighed and properly sized end screens.

(e) Label, preserve, and/or store samples as required.

Epoxy-stabilized cores can be frozen and drilled using the procedures described in a-e above or by the procedures described for plug samples (see 3.5.2.4.a).

2. Unstabilized cores—Unfrozen cores can be cut using a plunge cut technique. The cutter should be slowly pushed in the core and removed. The cut plug should be gently removed from the cutter and inserted directly into a preweighed sleeve. The plug ends are then trimmed.

3.6 CORE CLEANING

3.6.1 Introduction

Prior to most laboratory measurements of porosity and permeability, the original fluids must be completely removed from the core sample. This is generally accomplished through flushing, flowing, or contacting with various solvents to extract hydrocarbons, water, and brine.

3.6.2 Apparatus and Supplies

Various techniques and apparatus for cleaning are described in the procedures below and in Section 4.

Some solvents used for hydrocarbon extraction purposes are listed in Table 3-1. Listed solvents are those most frequently used for extracting samples for routine analysis. Some are preferred for specific applications; e.g., chloroform has been found to be excellent for many North American crudes and toluene has been found useful for asphaltic crudes. Prior to cleaning samples with unknown oil properties, a subsample should be tested with various solvents for cleaning efficiency.

Since residual salt crystals affect the measured porosity and permeability, core samples containing a formation water with high salinity may require additional extraction to remove

salt. Salt can be removed with methyl alcohol or other solvents in which salt is soluble.

The various solvents used for extracting core samples can be reclaimed by well-known physical and chemical methods. Such recovery techniques can make practical the use of an expensive solvent.

Table 3-1—Selected Solvents and Their Use

Solvent	Boiling Point, °C	Solubility
Acetone	56.5	oil, water, salt
Chloroform/methanol azeotrope (65/35)	53.5	oil, water, salt
Cyclohexane	81.4	oil
Ethylene Chloride	83.5	oil, limited water
Hexane	49.7–68.7	oil
Methanol	64.7	water, salt
Methylene chloride	40.1	oil, limited water
Naphtha	160.0	oil
Tetrachloroethylene	121.0	oil
Tetrahydrofuran	65.0	oil, water, salt
Toluene	110.6	oil
Trichloroethylene	87.0	oil, limited water
Xylene	138.0 –144.4	oil

3.6.3 Precautions

The following precautions should be enforced during core sample cleaning operations:

a. When using solvents, it is the responsibility of the user to establish appropriate safety and health practices prior to use and to comply with all applicable regulatory requirements regarding use and disposal of materials.

b. The solvent selected should not attack, alter, or destroy the structure of the sample.

c. Chloroform may hydrolyze during extraction, forming hydrochloric acid as a product.

d. Not all solvents are compatible with all extraction equipment configurations. Possible solvent/equipment reactions should be considered in selecting equipment and solvent for use in core cleaning operations.

e. Closed-type electrical heaters should be used whenever flammable solvents are used. Safety precautions such as adequate ventilation of the laboratory and ready accessibility of fire extinguisher equipment and safety showers should be observed.

f. Extraction should be conducted under ventilating hoods equipped with forced-draft ventilation.

g. Temperature effects on the samples should be considered. See 3.7 for recommendations regarding drying of core samples.

3.6.4 Procedures

3.6.4.1 Solvent Flushing by Direct Pressure

Extraction of hydrocarbons and salt from reservoir rocks can be achieved by injecting one or more solvents into the core sample under pressure and at room temperature. The pressure used should be dependent on the sample permeability and may range from 10 to 1,000 psi. The core samples may be held in a rubber sleeve under overburden pressure or in a suitable core-holding device that will permit the flow of solvent through the matrix of the sample. The volume of solvent required to completely remove hydrocarbons in the core sample is dependent on the hydrocarbons present in the sample and the solvent used. The core is considered clean when the effluent is clean. In some instances, more than one solvent may be required to remove heavy, asphaltic-type crude oils.

3.6.4.2 Flushing by Centrifuging

A centrifuge with a specially designed head is used to spray warm, clean solvent (from a still) against the core samples. The centrifugal force causes solvent to flow through samples displacing and extracting the oil (and water). The speed of rotation should be varied from a few hundred to several thousand revolutions per minute (rpm), depending on the permeability and degree of consolidation of the core. Most common solvents can be used. The apparatus and procedure are described in Conley and Burrows.[1]

3.6.4.3 Gas-Driven Solvent Extraction (U.S. Patent 2,617,719)

In this procedure, a core is subjected to repeated cycles of internal-dissolved or solution-gas drive until the core is cleaned of hydrocarbons. The remaining solvent and water are removed by means of a drying oven.

[1]Conley, F. R., and Burrows, D. B., "A Centrifuge Core Cleaner," *Journal of Petroleum Technology,* VIII, 61, October 1956.

When a core from an oil-bearing formation is brought to the surface and depressurized, the gas dissolved in the oil comes out of solution and displaces some of the oil and water out of the core. This results in some gas-filled pore space at atmospheric pressure. The gas-filled space in the core can be almost completely filled with solvent by surrounding the core with a suitable solvent containing a dissolved gas and applying sufficient hydraulic pressure. Under this condition, the solvent mixes with oil in the core and subsequent depressuring to atmospheric pressure removes some of the residual oil.

Carbon dioxide gas is excellent for this purpose because of low fire or explosion hazards and high solubility in most solvents. Some of the solvents that can be used are naphtha, toluene, or mixtures of solvents. With certain types of crude oil, cleaning time may be reduced if the core chamber is heated by a water bath, steam bath, or by electric heaters. One successful application of this method for routine cleaning of cores uses carbon dioxide and toluene at 200 psig, with a hydraulic pressure of 1,000 psig. Cycles of approximately 30 minutes are used. The apparatus and procedure are described by Stewart.[2]

3.6.4.4 Distillation Extraction Method

A Soxhlet extractor and suitable solvent or solvents should be used to dissolve and extract oil and brine. Extraction can be arranged in a manifold so that the oil-and water-laden solvent siphons from each extractor into a common still from which fresh solvent is continuously distilled, condensed, and again distributed to all the extractors.

The cleanliness of the sample is best determined from the color of the solvent that siphons periodically from the extractor. Extraction should be continued until the extract remains clear. Non-luminescence of extract under fluorescent light is a good criterion for determining complete extraction of the oil for a given solvent. It should be noted that the complete extraction of certain oils from core samples may require more than one solvent, and the fact that one solvent is clear after contact with the sample does not necessarily mean that oil has been completely removed from the sample. Details of one apparatus and procedure may be found in 4.3.

3.6.4.5 Liquefied Gas Extraction

Liquefied gas extraction uses a pressurized Soxhlet extractor and a condensed, low-boiling-point, polar solvent. The process is a distillation extraction procedure that uses pressurized solvent to clean the core. Solvent is regenerated through low temperature distillation. Because the extraction is conducted at or below room temperature, it can be performed on heat-sensitive core such as those containing gypsum.

[2]Stewart, Charles R., U. S. Patent Number 2,617,719, "Cleaning Porous Media," November 11, 1952.

3.6.5 Advantages

Cleaning a core removes original fluids from the core, preparing it for further tests that do not require those fluids.

3.6.6 Limitations

Individual test conditions may require a particular technique for optimal results. Some techniques may be more applicable to particular rock types or cleaning requirements.

3.7 DRYING

Conventional core samples can be dried by the methods listed in Table 3-2:

Table 3-2—Core Sample Drying Methods

Rock Type	Method	Temperature, °C
Sandstone (low clay content)	Conventional oven	116
	Vacuum oven	90
Sandstone (high clay content)	Humidity oven, 40% relative humidity	63
Carbonate	Conventional oven	116
	Vacuum oven	90
Gypsum-bearing	Humidity oven, 40% relative humidity	60
Shale or other high clay rock	Humidity oven, 40% relative humidity	60
	Conventional vacuum	

Each core sample should be dried until the weight becomes constant. Drying times may vary substantially, but are generally in excess of four hours.

3.7.1 Precautions

Some precautions that should be observed in drying samples for routine core measurements are:

a. Samples containing clays must not be dehydrated during preparation. Care must be exercised in drying these samples. In some cases, temperatures lower than those indicated in Table 3-2 should be used to prevent the dehydration of clays.
b. Samples containing gypsum will require specific procedures during preparation. Extreme care should be exercised to avoid both loss of water and change of the crystalline structure.
c. Samples must be protected from erosion by the drip of clean solvent when utilizing the distillation extraction technique.
d. The extraction technique should not physically damage the core.
e. The usual criterion for sample cleanliness is a clean extract. However, many solvents are not complete solvents for all types of oils and a clean extract may reflect oil solubility and not complete extraction.

f. Samples containing heavy asphaltic oils may require the cycling of more than one solvent.
g. More effective core cleaning can often be achieved by a combination of solvents.
h. Allow solvent laden samples to vent in a fume hood before placing in a closed drying oven.

3.8 SAMPLE PRESERVATION

Preservation of samples in the laboratory will depend on the length of time between tests and the type of tests to be done. Any storage or preservation technique should ensure that structural integrity is maintained and that unwanted drying, evaporation, and oxidation are avoided.

Laboratory samples can be preserved using the techniques described in 2.5. Freezing of samples in the laboratory should follow the precautions outlined in 2.4.4, 2.4.5, and 2.5.2.2.

SECTION 4—FLUID SATURATION METHODS

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4 Fluid Saturation Methods

4.1 INTRODUCTION

This section documents the specialized techniques developed for analysis of core samples with different sample physical characteristics and sizes, obtained by various coring methods. Table 4-1 lists the principal techniques for determining core fluid saturations.

Paragraphs 4.2 through 4.5 describe the seven principal saturation analysis methods listed in Table 4-1 for full-diameter cores and plugs/sidewall cores. Plugs are defined as samples drilled or shaped from cores recovered with any of several full-diameter coring devices. Sidewall cores are defined as cores that are obtained after the wellbore has been created by the drill bit or some type of full-hole coring device.

Paragraphs 4.6 through 4.8 address fluid saturation analysis methods for coal, oil shale, and gypsum-bearing samples. Oil shale and coal samples may be obtained with full-diameter coring devices. However, oil shale and coal may also be quarried from surface or shallow formations, and require non-routine sampling procedures different from those typically used for reservoir core. The reporting format of fluid saturations for these latter materials differs from typical reservoir rocks in that the saturations may be expressed as weight percent or gallons per ton instead of percent of pore volume.

Scanning methods that are capable of measuring fluid saturations by non-destructive techniques are included in 4.5. Additionally, a historical section (see 4.9) is included in which some previously acceptable methods for fluid saturation determination are described, but are not recommended.

A difficult, if not impossible, formation evaluation task is recovering a core having the same saturation and distribution of fluids as occurred in the formation prior to coring. Changes in fluid content and distribution frequently take place during the coring, retrieval, preservation, and transportation phases. Laboratory handling, sampling, and testing processes can bring about further alterations. These issues are not addressed in detail in this section, but the reader should be aware of the many complications that influence the final fluid saturation values furnished in a laboratory core analysis report. Fluid saturation analysis may alter the wetting of the core and thus affect further analyses on the core material.

Table 4-1—Rock Type and Recommended Fluid Saturation Testing Method(s)

Rock Type	Recommended Testing Methods
Consolidated clastics, carbonates	a,b,c,d,e,f
Unconsolidated (light oil)	c,d,e
Unconsolidated (heavy oil)	c, c(*),e
Vuggy carbonates	b,d,e,f
Fractured	a,b,d
Clay bearing	a,c(*),e
Evaporites	g,e
Low permeability	a,b,c,d,e,f
Coal	h
Shales	a,b,c
Oil shale	a(*)
Diatomite	c,e

Legend:
a = retort at atmospheric pressure.
b = distillation extraction (full diameter).
c = distillation extraction (plug).
d = pressure-retained core method.
e = solvent flushing/Karl Fischer.
f = sponge core barrel method.
g = gypsum-bearing method.
h = coal method.
(*) = modified procedure.

4.2 RETORT METHOD AT ATMOSPHERIC PRESSURE

4.2.1 Basic Procedure

4.2.1.1 Principles of Analysis

Oil and water fluid saturations are obtained by a high-temperature retorting process in which the oil and water contained in a fresh sample of crushed core material are vaporized, condensed, and collected in calibrated glassware. The gas saturation is determined on an adjacent, lithologically similar sample by placing it in a mercury pump and measuring the amount of mercury injected with the water and/or oil in place.

4.2.1.2 Apparatus

The following items describe suitable apparatus used in the basic retort method:

a. Stainless steel retort(s)—See Figure 4-1. Such retorts are designed to hold 100 to 175 grams of crushed core material. Each retort is equipped with a threaded cap, that in turn is equipped with a gasket to prevent leakage of condensable gases. A long, stainless steel condensing tube is connected to the opposite end of the retort. A coarse mesh screen is placed in the bottom of the retort to prevent crushed core material from entering the condensing tube.

b. Oven—See Figure 4-1. Such ovens are capable of holding multiple retorts. Strip-type electrical heating elements are preferred. A temperature controller, thermocouple, and temperature readout device are also required.

c. Water bath—See Figure 4-1. A water bath should be located so that the condensing tubes pass through it, thereby enhancing condensation of the evolved gases.

d. Calibrated glass receiving tube—A common 15-ml centrifuge tube is generally acceptable, although a tube with a larger capacity may be required. The required tube size depends on the amount of retorted material and the total fluids contained within that material. The receiving tube is affixed to the end of the condensing tube with a rubber stopper arrangement.

e. Rock hammer and crusher—A geologist's pick or a masonry hammer may be used. Typical crushers are of the "chipmunk" variety.

f. Diamond saw—A variety of commercial core saws, equipped with diamond saw blades, may be used.

g. Mercury pump—Typical mercury pumps are equipped with a cell, lid, and valve arrangement. Typical cells can accommodate a sample with a bulk volume of 10 to 15 cm^3. The mercury pump is equipped with a vernier caliper graduated in 0.1 cm^3 increments. A pressure gauge capable of indicating pressures to 1,000 psi (6.895 MPa) is connected to the system.

h. Centrifuge—A hand-crank centrifuge capable of holding four of the 15-ml centrifuge tubes is suitable. Other centrifuges commensurate with the tube size and centrifugal forces required may be used.

4.2.1.3 Procedures/Precautions

The basic procedure is normally conducted on samples taken from full-diameter cores. Typical analyses are done on a foot-by-foot basis, except some lithologies such as shale, anhydrite, gypsum, and heavy clays are usually described and skipped in this type of analysis. A representative sample approximately two inches in length is broken from each foot using a geologist's or similar type hammer. This sample is broken lengthwise into two halves. One half is designated for the fluid saturation sample and the other half is used to obtain a plug for the determination of permeability and/or Boyle's Law porosity (see 5.3.2.1.1 and 5.3.2.2.1). Alternatively, a 1 to 2 inch (25.4 to 50.8 millimeter) piece may be broken adjacent to the piece selected for fluid saturation testing and the plug drilled from it. The advantage of this latter sampling method is that it can afford a larger and longer plug, thereby reducing the potential for drilling fluid contamination.

The sample (10 to 15 grams) for the gas space measurement should be chipped or shaped to a convenient size, i.e., dimensions that allow it to be placed in the cell of a mercury pump. In addition, the sample should be well rounded to permit mercury conformance. The weight of the sample is recorded and the bulk volume of this sample is determined by mercury displacement for later use in the calculation of a summation-of-fluids porosity (see 5.3.2.2.2 and 4.2.1.4). The cell valve is closed and the pressure increased to 750 to 1,000 psi (5.171 to 6.895 MPa) depending upon the permeability and/or porosity of the sample. The amount of mercury injected (properly corrected by pump calibration procedures, see 4.2.1.8) is recorded.

The sample for the water and oil measurement should be crushed into approximately $^1/_4$-inch (6.4-centimeter) pieces. The crushed core material is screened using a 3 or 4 Tyler mesh screen [approximately 0.25 inch (6.4 millimeter)] to remove any fines created in the crushing process. An amount of this material (usually 100 grams to 175 grams) is weighed to the nearest 0.01 gram and poured into the retort. The retort lid is secured and the retort, along with other similarly prepared retorts, is placed in the oven. The oven temperature is initially maintained at 350°F (177°C) (Hensel, 1982[1]) until all samples cease to give up water. The water volumes are recorded and the oven temperature is then raised to a temperature between 1,000 and 1,200°F (538 and 649°C). The fluid collection tubes are monitored and when all samples cease to give off fluids the process is considered complete. The retorting time usually ranges from 20 to 45 minutes. The total water and oil volumes are recorded.

The initial oven setting of 350°F (177°C) is selected to remove pore water, absorbed water, interlayer clay water (e.g., smectite), and waters of hydration (e.g., $CaSO_4$), but not hydroxyl clay water. However, this method is not appropriate for samples containing gypsum or large amounts of montmorillonite. Gypsum bearing core analysis procedures are described in 4.8 as a special core analysis procedure. Although samples containing montmorillonite can pose problems in determining accurate fluid saturation, heating to 350°F (177°C) is documented (Brown, 1961[2]; Mackenzie, 1970[3]) as the lowest temperature at which hydroxyl waters are desorbed. Although the clay hydroxyl water weight loss at this temperature may be as small as 5 percent, it may impact the final water and oil saturation values. This method is superior to other retorting techniques, e.g., the plateau method (Hensel, 1982[1]). The plateau method suggests that the plotting of water collected versus time data, while continuous heat is applied, will yield plateaus whereby a proper time for reading only pore water can be determined for each formation being tested.

The final retorting temperature of 1,000 to 1,200°F (538 to 649°C) may yield incorrect oil volumes. Some core material may contain solid hydrocarbons, e.g., kerogen, gilsonite, etc. that break down at these temperatures. The analyst should take mineral composition into consideration and may elect to make arbitrary and judicious corrections for the oil collected. In extreme cases, the retort procedure should not be used and other methods, e.g., distillation extraction (Dean-Stark) (see 4.3) procedures should be employed.

Other sources of error in the measurement of oil volumes stem from: (a) mechanical hold-up in condensing tubes, (b) leaking retort seals, and (c) coking and cracking of certain

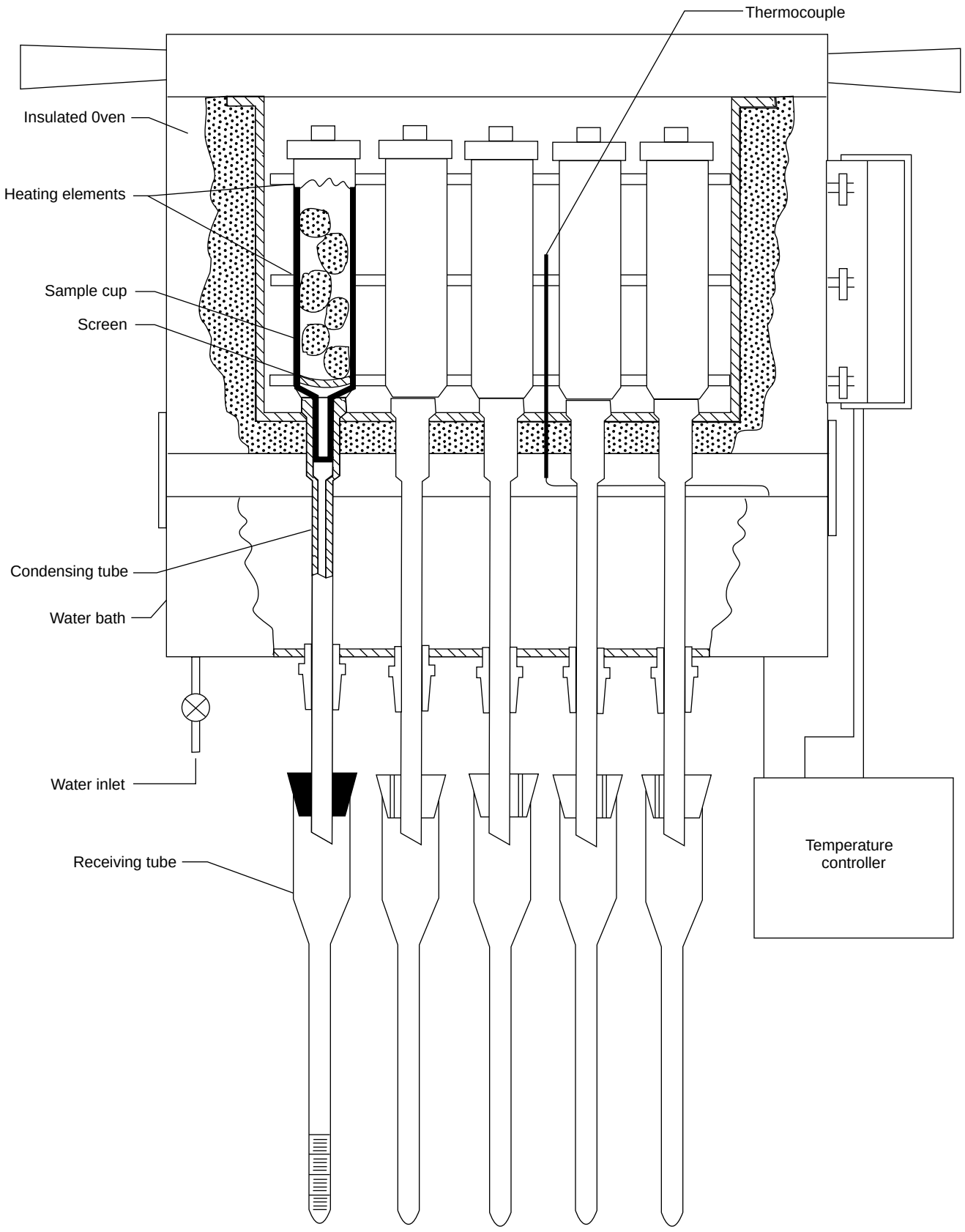

Figure 4-1—Stainless Steel Oven Retort and Water Bath—Atmospheric Pressure

oils. Oil volume correction curves (See Figure 4-2) must be employed routinely.

The "total" water collected after the complete process may be of value in determining if "pore" water may have been mechanically retained in the condensing tube. The analyst may note a calculated grain density for a sample that is anomalous to the general trend. A comparison between the initial (pore) water volume and the final (total) water volume indicates a difference greater than expected based on similar water volume differences from adjacent samples. Laboratory personnel may then elect to make judicious corrections to the initial water volumes so that reasonable data may be obtained. The reporting of obviously anomalous data is not in the best interest of the end user. Such corrected data should be flagged and a footnote inserted.

Crushed core material is vulnerable to fluid loss due to evaporation, particularly of water, due to the high surface area of the crushed core. Exposure time for the crushed core material should be minimized.

Note: Since handling mercury is required for this fluid saturation method all existing safety and health standards should be followed. Consult relevant environmental regulations and local laws.

4.2.1.4 Calculations

The corrected oil (See Figure 4-2) and pore water collected for each sample along with the gas volume may be expressed as a percentage of bulk volume as follows:

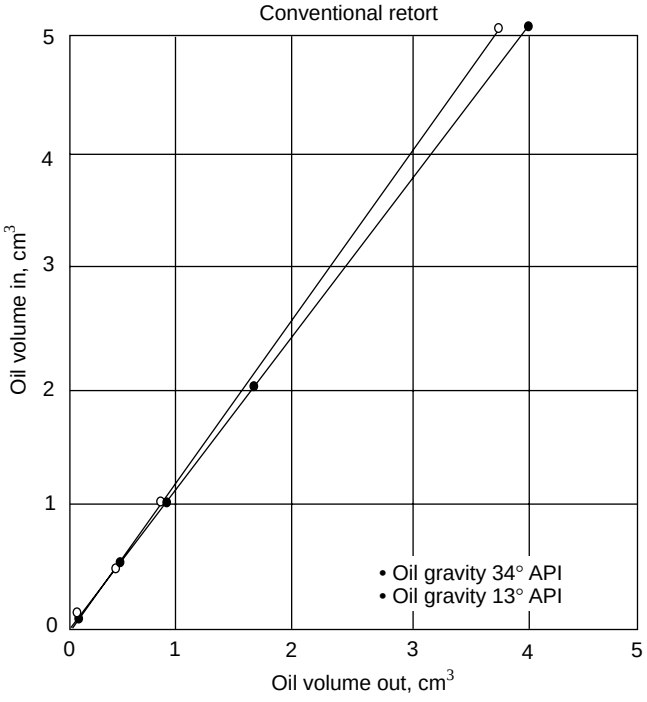

Figure 4-2—Retort Oil Correction Curve

$$NDHg = WtHg/BvHg$$

$$BvRet = WtRet/NDHg$$

$$GB = (HgInj \times 100)/BvHg$$

$$OB = (OilVol \times 100)/BvRet$$

$$WB = (WatVol \times 100)/BvRet$$

$$GS = (GB \times 100)/(GB + OB + WB)$$

$$OS = (OB \times 100)/(GB + OB + WB)$$

$$WS = (WB \times 100)/(GB + OB + WB)$$

Where:

NDHg = natural density of mercury pump sample, g/cm^3.
WtHg = initial weight of mercury pump sample, g.
BvHg = bulk volume of the mercury pump sample, cm^3.
WtRet = weight of the retort sample, g.
HgInj = volume of injected mercury corrected by pump calibration factors, cm^3.
OilVol = volume of recovered oil corrected with oil calibration curves, cm^3.
WatVol = volume of initial water recovered, cm^3.
BvRet = bulk volume of the crushed retort sample, cm^3.
GB = gas saturation as a percentage of bulk volume.
OB = oil saturation as a percentage of bulk volume.
WB = water saturation as a percentage of bulk volume.
GS = gas saturation as a percentage of pore volume.
OS = oil saturation as a percentage of pore volume.
WS = water saturation as a percentage of pore volume.

Grain density may be calculated from the data obtained during the testing procedure (Hensel, 1982[1]). These grain density data are of primary value as a means of quality control to ensure that erroneous readings have not been taken, that all fluids have been determined properly, and no calculation errors have been made. This is not the most accurate means of determining the true grain density of the rock material; however, the values obtained should be reasonably representative of the lithology being tested. Thus, the grain density calculation may be considered as a qualitative data evaluation tool.

4.2.1.5 Advantages

Advantages of the basic retort method include:

a. Fluids are collected from relatively large samples ensuring better representation of the lithology and increasing the potential precision of all measurements.
b. The analytical process is rapid and provides required saturation data within hours. Many samples can be evaluated in a short time and in a cost-effective manner, if sufficient retorts and ovens are available.
c. Fluid volumes are measured directly as opposed to other analytical techniques that rely on combinations of fluid volumes and total weight loss.

d. Grain loss, commonly associated with friable sandstones and some carbonates, does not affect the fluid saturation data.

4.2.1.6 Limitations

Limitations of the basic retort method include:

a. Water saturation (and porosity) may be too high if samples contain large amounts of montmorillonite or gypsum that break down at high temperature. This in turn will result in oil saturations that are too low since the volume of oil would be expressed as a percentage of a pore volume that is too high.

b. Oil saturation (and porosity) may be too high if the sample (e.g., some shales) contains solid hydrocarbons that break down at high temperature. This in turn will result in water saturations that are too low since the pore water is expressed as a percentage of a pore volume that is too high.

c. Oil volume correction curves are required and it may not be feasible to obtain the corrections for the oil in question. It may be necessary to use generalized correction curves, based on reported or assumed API gravities.

d. A second piece of core material is required to determine bulk volume, natural density, and gas-filled pore volume. This sample must be lithologically similar to the sample that is crushed for the oil and water data.

e. The distilled liquids may form emulsions.

4.2.1.7 Accuracy/Precision

Accuracy/precision of the basic retort method is:

a. By using an oil volume correction curve, the accuracy of the oil value obtained is within ±5%, and reproducibility is within ±2.5% of measured volumes.

b. The accuracy of the water volumes obtained by this method is ±2.5% of measured volumes.

4.2.1.8 Calibration

Calibration issues for the basic retort method include:

a. Graduated glassware should be checked for accuracy by using a microburette with water. The cleanliness of the glassware could impact readings due to a lack of complete wetting and indistinct meniscus.

b. The mercury pump used for bulk volumes should be calibrated with steel blanks of known volumes. Additionally a pump/system factor should be determined at the pressure used to determine the gas-filled pore volume. Correction curves and/or equations should be used in both instances.

c. All balances should undergo routine maintenance.

d. Retorts should be regularly subjected to vacuum tests to ensure the seals are performing properly.

4.2.2 Percussion Sidewall Cores

4.2.2.1 Principles of Analysis

The method is a modification of the basic retort procedure (see 4.2.1). The main difference is that gas, oil, and water volume measurements are made on the same sample as opposed to the basic procedure where adjacent core pieces are required. Smaller samples are involved in the sidewall procedure and therefore the equipment is scaled down to provide comparable accuracy. This method may be applied to rotary sidewall cores, but damages the samples for further testing.

4.2.2.2 Apparatus

Suitable apparatus for retorting of sidewall cores include:

a. Stainless steel retorts—See Figure 4-3. The overall design is similar to the retorts used in the basic procedure, i.e., the retort is composed of a cup section that holds the sample, a gasket/lid arrangement, and a long condensing tube. The cup section is greatly reduced in size. A bottle cap may be substituted for a screw-on lid, such cap being affixed with a standard capping device. The condensing tube is shorter and its outside and inside diameters smaller than the tubes used for the large retorts.

b. Oven—The sidewall oven is composed of the same components as the oven used in the basic procedure (see 4.2.1.2, b). The sidewall oven is tailored to accommodate multiple sidewall retorts.

c. Calibrated glass receiving tubes—The 15-ml centrifuge tube is not recommended, rather the receiving tube should be designed to accommodate very small fluid volumes (e.g., a maximum of 5 ml). The graduations should be such that readings/interpolations to the nearest 0.01 ml are possible.

d. Mercury pump—The same mercury pump used for the Basic Procedure (see 4.2.1.2, g) may be used, however a small sample chamber will enhance the bulk volume and gas space measurements.

e. An apparatus capable of piercing the jar lid is optional (see 4.2.2.3).

f. A hot wire gas detector capable of a qualitative measurement of combustible gases is optional (see 4.2.2.3).

g. A gas chromatograph is optional (see 4.2.2.3).

4.2.2.3 Procedures/Precautions

4.2.2.3.1 Procedures

Samples should be received in the laboratory sealed in glass jars with screw-top lids. The samples should be arranged in descending depth order. Prior to opening the jar many laboratories employ a procedure that involves puncturing the jar lid and making a measurement of the contained gases. This procedure will not be discussed in detail in this section, however this optional procedure measures combustible gases with

a hot wire detection device and some laboratories determine the head space gas composition by chromatography.

The sample recovery (sample length), while it is still in the jar, is noted by calibration marks on the side of the jar. Each sample is then removed from the jar and is cleaned of drilling fluid solids by using a single edge razor blade or utility knife. A brief description of the sample is recorded including rock type, grain or crystalline size, siltiness, shaliness, fossils, color, intensity, and distribution of fluorescence, and other megascopic lithological features. The sample is weighed to the nearest 0.01 gram.

Some laboratories may divide the prepared sample so as to provide a portion for a permeability and/or other measurements, e.g., particle size distribution, x-ray diffraction, etc. Obviously this is limited to initial samples of large size, e.g., 2 inches (50.8 millimeters) in length. Even so the fluid saturation data are compromised by testing a smaller sample. Most laboratories employ an empirical approach to sidewall permeability data. Neither of these practices will be discussed in this section.

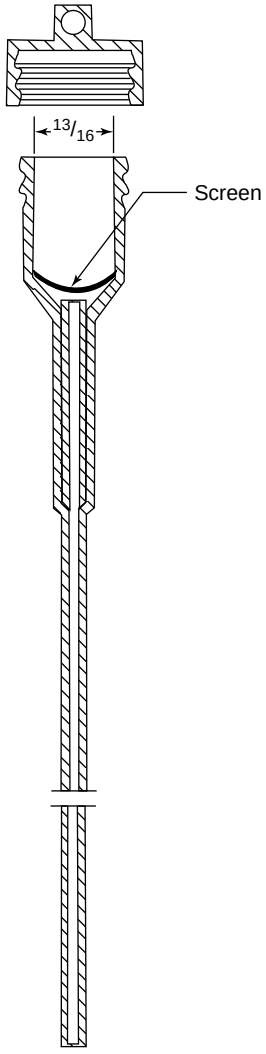

Figure 4-3—Retort Cup—Sidewall Sample

The sample is placed in the cell of a precalibrated mercury pump, and bulk volume is determined by mercury displacement (see 5.2.1). With the sample immersed in mercury, a valve is closed and the pressure raised to 750 psi (5.171 MPa). In tight (low permeability) samples, the pressure is raised to 1,000 psi (6.895 MPa). This procedure measures the gas-filled pore volume assuming that the compressibility of the water and/or oil present in the other pore spaces is minimal.

The sample is removed from the cell and broken into smaller pieces to observe the mercury penetration pattern. The penetration pattern is helpful in predicting probable production.

The retorting procedure described in 4.2.1.3 is followed, including the recording of initial (pore) and total water collected and the use of oil volume correction curves (see Figure 4-2).

4.2.2.3.2 Precautions

Sidewall core material is subject to fluid loss due to evaporation so exposure time to room conditions should be minimized.

Note: Since handling mercury is required with this fluid saturation method all existing safety and health standards should be followed. Consult relevant environmental regulations and local laws.

4.2.2.4 Calculations

The oil, pore water, and gas-filled pore volume may be expressed as percentage of bulk volume as follows:

$$GB = (HgInj \times 100)/BV$$

$$OB = (OilVol \times 100)/BV$$

$$WB = (WatVol \times 100)/BV$$

$$GS = (GB \times 100)/(GB + OB + WB)$$

$$OS = (OB \times 100)/(GB + OB + WB)$$

$$WS = (WB \times 100)/(GB + OB + WB)$$

Where:

BV = bulk volume of the sample, cm^3.
$HgInj$ = volume of injected mercury corrected by pump calibration factors, cm^3.
$OilVol$ = volume of recovered oil corrected with oil calibration curves, cm^3.
$WatVol$ = volume of initial recovered water, cm^3.
GB = gas saturation as a percentage of bulk volume.
OB = oil saturation as a percentage of bulk volume.
WB = water saturation as a percentage of bulk volume.
GS = gas saturation as a percentage of pore volume.
OS = oil saturation as a percentage of pore volume.
WS = water saturation as a percentage of pore volume.

Grain density may be calculated from the data obtained during the testing by slight modifications to the method proposed by Hensel, 1982.[1]

The bulk density, sometimes used in various laboratories, may be calculated by dividing the initial (whole) sample weight by the bulk volume.

4.2.2.5 Advantages

Advantages of the sidewall retort procedure include:

a. Gas, oil, and water measurements are made using one sample.
b. The method is relatively rapid.
c. This is a "direct" measurement method where gas, oil, and water measurements are made independently.

4.2.2.6 Limitations

Limitations of the sidewall retort procedure include:

a. The distilled liquids may form emulsions.
b. Oil and water calibration is required.
c. Volumes of oil <0.1 ml are difficult to determine accurately.
d. For very friable or unconsolidated sands the measured gas-filled pore volume may be high due to sample expansion when overburden pressure is released.
e. Samples must be handled carefully to minimize loss of liquids, especially water. In a low humidity atmosphere, water loss may be substantial.
f. Porosity and fluid saturation values can be in error if gypsum or hydratable clays (e.g., montmorillonite) are present and precautions are not taken in recording only "pore" water.
g. Calibration of all equipment is critical because samples are typically small and may contain equally small amounts of oil and water. Minute differences in recorded fluid volumes may result in significant ranges of saturation and porosity values.

4.2.2.7 Accuracy/Precision

Accuracy/precision for the sidewall retort procedure is:

a. The method's accuracy is strongly dependent on sample size, especially when determining oil volumes.
b. Analytical discretion should be used on testing samples with an initial weight of less than 3 grams.
c. By using oil volume correction curves, the precision of the oil value obtained is within ±5 percent of the measured volumes.
d. The precision of the water volumes is within ±3 percent of the measured volumes.

4.2.2.8 Calibration

Refer to 4.2.1.8 of the basic retort procedure.

4.3 DISTILLATION EXTRACTION METHOD (DEAN STARK)

4.3.1 Plug Samples

4.3.1.1 Principles of Analysis

This procedure is appropriate for plug samples and for rotary sidewall cores. The distillation extraction (Dean-Stark) method of determining fluid saturation depends upon the distillation of the water fraction, and the solvent extraction of the oil fraction from the sample. The sample is weighed and the water fraction is vaporized by boiling solvent. The water is condensed and collected in a calibrated receiver. Vaporized solvent also condenses, soaks the sample, and extracts the oil. The sample is oven dried and weighed. The oil content is determined by gravimetric difference.

4.3.1.2 Apparatus and Reagents

The following apparatus is suitable for this method and is illustrated in Figure 4-4. The apparatus should be set up in a fume hood or room suitable for exhausting solvent vapors.

a. General—The apparatus consists of an electric mantle or heating device with thermostatic controls. The distillation/extraction unit for one sample consists of a boiling flask, thimble, trap or calibrated sidearm, and condenser.
b. Flask—The flask is wide-mouthed, long-necked, and may contain indentations at the base of the neck to support the extraction thimble.
c. Trap—The trap or sidearm has a graduated section marked in 0.1 ml divisions. The graduated trap can be made smaller or larger to accommodate samples with very small or very large water volumes. Attached to the graduated section is a glass tube or arm that is bent at a right angle with a glass joint on the end. The glass joint has a drip tip molded on it and the opening in the tip should be designed so that the dripping solvent is directed in the center of the flask to ensure soaking of the core plug below. A modification to the trap (Figure 4-5) enables the water to be determined gravimetrically.
d. Condenser—The condenser is a water-cooled, reflux, glass-tube type, with a condenser jacket approximately 11.8 inches (300 millimeters) long and an inner tube (see Figure 4-4). The bottom is fitted with a drip tip and the inside tube should be vertical to reduce the difficulty of removing water from the surfaces of the condenser and trap.
e. Desiccant holder—A glass tube desiccant holder is fitted to the top of the condenser when samples are being extracted and a rubber stopper is used when the apparatus is idle.
f. Extraction thimbles—A glass thimble with a fritted glass bottom is recommended for holding a plug sample. Alternatively, a cellulose thimble may be used. However, types of thimble materials other than glass may cause errors in weighing because of absorption of atmospheric water. Also, thimbles made of glass allow viewing of the plug to ensure

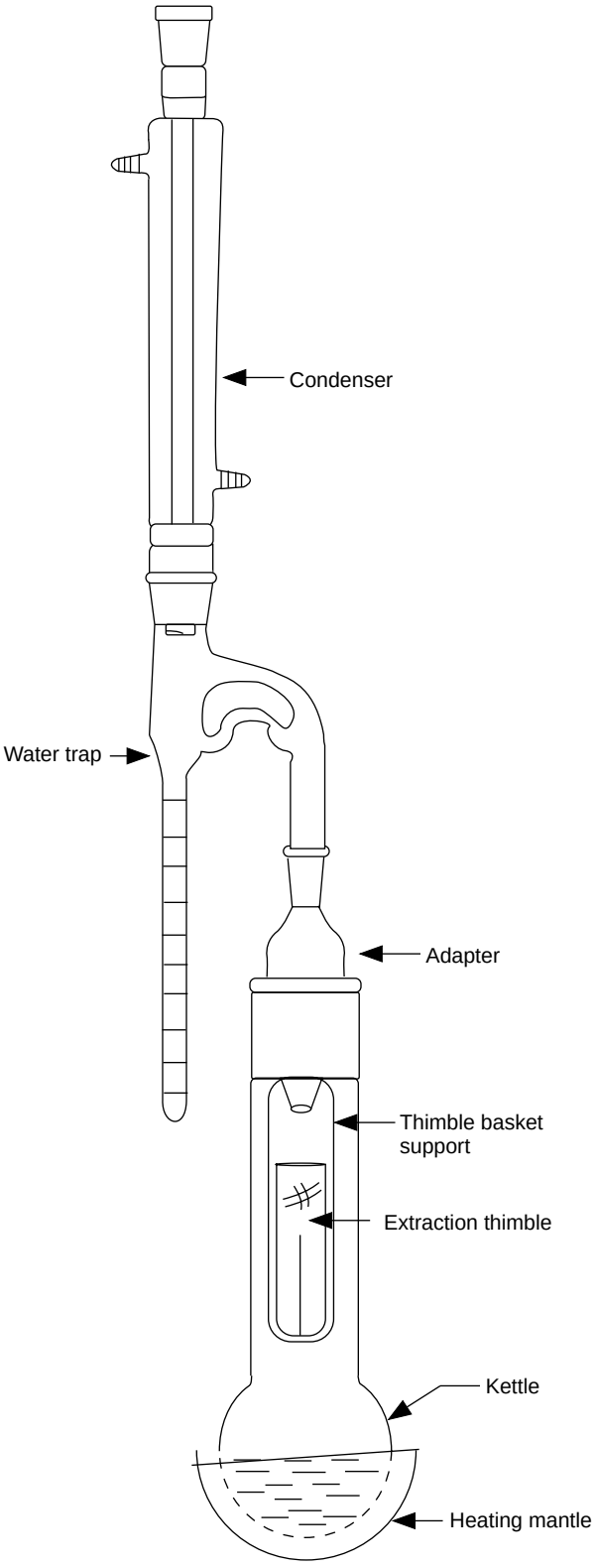

Figure 4-4—Dean-Stark Apparatus for Volumetric Determination of Water

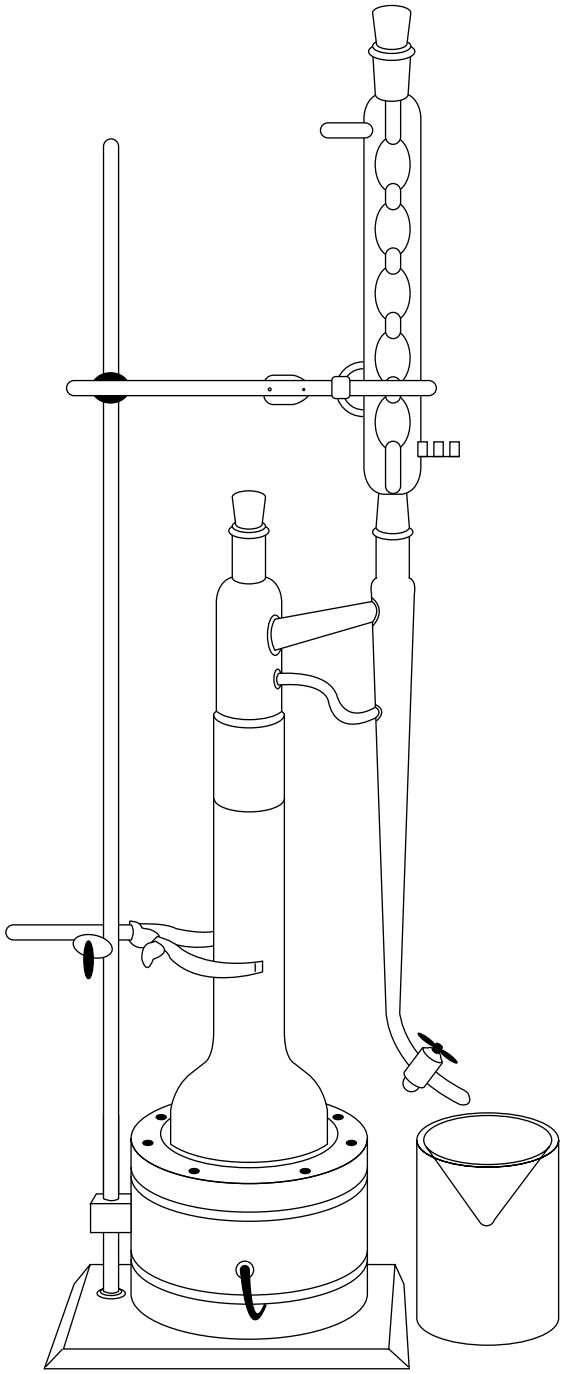

Figure 4-5—Dean-Stark Extraction Apparatus for Gravimetric Determination of Water

that the solvent is dripping and soaking the sample for thorough extraction. A small piece of glass wool may be used to cover the core plug to prevent erosion by the solvent reflux from the condenser.

g. Extraction cup—Cups fitted with siphons can be used to hold the thimbles and allow alternating immersion/drainage cycles. This can increase the efficiency of the extraction process.

h. Drying oven—A convection or vacuum oven may be used having a temperature control of ±2°C. An explosion-proof model is preferred, but non-explosion proof models can be used. For samples containing large amounts of montmorillonite and other clays, a humidity oven can be used to help preserve a single or double water layer to better approximate the expected hydration state of the clays in the reservoir. (Humidity levels during drying procedures are important in maintaining proper waters of hydration.)

i. Boiling chips—Glass beads or small chips of alundum material may be placed in the bottom of the boiling flask. These aid in reducing the tendency of the solvent to "bump" as it becomes more saturated with oil. If bumping or super heating occurs it creates an undesirable boiling process, contaminating the sample and extending the time required for the initial phase of the distillation extraction process.

j. Solvent—Toluene (reagent grade) or other suitable solvent.

4.3.1.3 Procedures/Precautions

4.3.1.3.1 Procedures

The samples are prepared using a fluid that is compatible with the fluid used to cut the whole core or drilled sidewall cores in the well. This ensures the preservation of residual oil or water saturation depending on the use of oil or water-based fluid. In the case of frozen oil sands samples, liquid nitrogen should be used. The excess fluid remaining on the sample after the trimming of the ends should be wiped off using a material that will remove the surface fluid but will not pull fluid from the interior in a sponge-like manner. This process is best described as a squeegee action. The sample's surface should appear dry (with a dull luster) and not shiny with excess fluid. The thimble/plug sample should be weighed to the nearest milligram (0.001 gram) on an analytical balance. The process should be accomplished quickly to minimize evaporation of the fluids in the sample. After final weighing, the samples and/or thimbles should be placed in the apparatus immediately, or should be stored in a container to prevent evaporation until they are placed in the apparatus. This storage period should be kept to a minimum. The necessity for weighing each component is to ensure complete accountability for weight loss. When extraction thimbles are used they should be at room temperature and thoroughly dry.

The samples are extracted with a solvent that does not donate to or absorb any of the collected water. Solvent conditioning can be provided by adding at least 1 percent water by volume to the solvent and pre-boiling the solvent until the water concentration is stable. Some laboratories add 15 percent water by volume to the solvent. Water should be in excess of the amount required to bring common solvents into equilibrium when the water is boiled away.

The desiccant trap ensures that atmospheric moisture (humidity) does not affect the water collected.

Water can adhere to the condenser and to the side of the trap. This fluid may cause appreciable error, particularly for a plug sample of low porosity. The adhered water is dislodged with a stiff solvent stream from a laboratory squirt bottle or a small gauge wire with a very small loop at the end. The wire is used to physically dislodge water particles that are not removed by the stream of solvent. Detergent is sometimes used to flatten the solvent/water interface for improved accuracy; but the use of detergent is not generally recommended because the effects of three phase fluid interaction are unpredictable and the wettability of the sample will be altered.

The distillation/extraction process continues for a minimum of 48 hours. Water levels should be monitored daily and the process stopped only when there is no change in volume of water recovered in 24 hours. Longer times may be required depending on the size of the plug sample and its permeability. This is to ensure that the solvent used has extracted all the oil possible from the plug material. Where heavy oil (low-gravity, high-asphaltene) is involved, another solvent type (see 3.1) is required to thoroughly clean the plug. A complete record of the volumes of water collected should be noted. The rate of extraction and efficiency may be increased by using extraction cups equipped with siphons. Alternative methods that speed extraction involve using the described apparatus until the amount of water collected is at a stable end point, and then doing one of the following with the samples and the thimbles:

a. Place the samples in a vapor phase unit to complete the oil extraction.

b. Place the samples in a CO_2-toluene pressure core cleaner to complete the oil extraction. This should be restricted to competent, low-permeability samples that will not be physically altered by this process.

c. Place the sample in a Soxhlet extractor for alternating immersion and drainage type oil extraction.

d. Place the sample in a flow-through oil extraction apparatus.

e. Alternate between solvent types (e.g., toluene and methanol).

The extraction efficiency is evaluated by treating the sample with chlorothene under an ultraviolet light source to determine if there is any remaining oil, that fluoresces, or by measuring the grain density of the sample. If the grain density is lower than anticipated for the rock type, the sample may need further extraction.

The sample/thimble should then be dried to a stable weight. Samples with high saturations of flammable solvents should be dried in an explosion-proof convection or vacuum oven, unless the excess solvent is allowed to evaporate before being put into the oven. This avoids a potential explosion or fire situation. When the sample/thimble is dried, it should be allowed to cool to room temperature in a sealed container such as a desiccator and the weighed. An oven that adds moisture to the sample should not be used for this step. However, when porosity and permeability will be measured, samples containing large amounts of montmorillonite and other clays may need to be dried in a humidity oven to preserve the hydrated state found in the reservoir. (The humidity level in the oven is important in maintaining proper waters of hydration for measuring permeability and porosity.)

Errors occur by not accounting for a chipped thimble, the core losing small particles, and/or the loss on the sample surface of precipitated salt from concentrated brines. In the case where heavy salt precipitation occurs, the salt is removed from the sample by extraction with methanol or similar solvent. The sample is then dried and weighed. The weight of water collected in the trap is subtracted from the total liquid weight loss to determine the oil weight extracted from the plug sample.

Proper water flow in the condenser will maintain a condenser temperature sufficiently cool so that the vapors condense in the bottom third of the condenser column.

4.3.1.3.2 Precautions

Local regulations regarding the safe use of reagents should be followed. In general, however, the following safety considerations should be noted:

a. Oil may contain compounds exhibiting carcinogenic properties. It may be flammable.

b. Toluene is moderately toxic by skin absorption and inhalation. It possesses irritant and anesthetic properties and is highly flammable.

c. Many other solvents, while effective for the oil removal process, may be dangerous and toxic. Consult relevant environmental regulations and local laws governing the use of these solvents.

d. Solvent vapors should condense in the lower third of the water-cooled heat exchanger on top of the water trap.

e. The analyst should be aware of the change in solvent boiling point with altitude or the change in the water boiling point due to salt in solution. The solvent's boiling point should be checked to ensure an adequate temperature for water distillation. When KCl drilling fluids are used, the filtrate water will have salt concentration on the order of 300,000 ppm, and, consequently, boil at a much higher temperature than fresher water. The use of orthoxylene is suggested as a substitute for toluene in these cases.

4.3.1.4 Calculations

The following calculations are appropriate for this method:

$$\text{Weight \% Water (Gravimetric)} = \frac{(\text{Weight of Water}) \times 100}{(\text{Initial Sample Weight})} \quad (1)$$

or

$$\text{Weight \% Water (Volumetric)} = \frac{(\text{Volume of Water}) \times (\text{Density of Water}) \times 100)}{\text{Initial Sample Weight}}$$

$$\text{Weight \% Solids (Gravimetric)} = \frac{\text{Dry Weight of Sample} \times 100}{\text{Initial Sample Weight}} \quad (2)$$

$$\text{Weight \% Oil (Gravimetric)} = \frac{(\text{Initial Weight} - \text{Dry Weight} - \text{Weight of Water}) \times 100}{\text{Initial Weight of Sample}} \quad (3)$$

The saturations are normally expressed as percentages of the sample pore space. Therefore, the sample porosity, water density, and oil density are required. If the connate water is a highly concentrated salt solution, the water density must be corrected for the salt in solution.

The following calculations apply:

$$\text{\% Water} = \frac{\text{Volume of Water} \times 100}{\text{Pore Volume}} \quad (4)$$

$$\text{\% Oil} = \frac{(\text{Weight of Oil})/(\text{Density of Oil}) \times 100}{\text{Pore Volume}} \quad (5)$$

The liquid content of the sample is reported to the nearest 0.1 percent of the pore space; e.g., 22.1 percent oil and 43.7 percent water.

Knowing the brine salinity and density, one can calculate volume of brine that was in the core from the volume of distilled water recovered as follows:

$$Vbr = [(Vw)(\rho_w)/\rho_b]\,[1{,}000{,}000/(1{,}000{,}000 - Cs)] \quad (6)$$

Where:

Vbr = volume of brine corresponding to the volume of distilled water collected from the plug, cm^3.

Vw = volume of distilled water collected from the plug (e.g., Dean-Stark), cm^3.

ρ_w = density of distilled water, g/cm^3.

ρ_b = density of lease brine having a concentration C_s of salt, g/cm^3.

C_s = concentration of dissolved salts in lease brine, =

$$\frac{(1{,}000{,}000)(\text{Weight of Salt})}{(\text{Weight of Brine})}\text{ppm}$$

4.3.1.5 Advantages

Advantages of this method include:

a. Water volume determinations are generally very accurate.

b. Typically, the sample is not damaged and can be used for further testing. However, the wettability may be altered and certain clays (e.g., montmorillonite) or gypsum may also be subject to change.

c. Relatively low temperatures [212°F (100°C)] are used; hence, little if any of the clay hydroxyl waters are removed.

d. The procedure is simple and requires little attention during distillation.

4.3.1.6 Limitations

Limitations of this method include:

a. Inaccuracies arise in the water determination due to the following:

1. Atmospheric water condenses in the condenser when atmospheric humidity is high. Desiccant tubes can be used to avoid the problem.

2. Water evaporates from the sample at room temperature when it is not immediately set up in the extractor with the condenser water circulating.

3. Water droplets stick to unclean glass in the sidearm or condenser.

4. Salt may precipitate inside the sample from connate brines (saline interstitial water). This can result in significant changes in porosity and/or permeability. Salt can be removed from the sample with methanol extraction.

5. Correction for the higher density of salt water is required when the total solids concentration exceeds 20,000 ppm (see 4.3.1.4, Equation 5).

6. Incomplete drying of solvents.

7. Loss of water due to the joints of the extraction flask not being vapor tight, or from too high an extraction temperature, or from insufficient water flow in the condenser.

8. The density of air must be considered for buoyancy only when the sample is weighed to the nearest 0.1 mg.

9. Extraction time may be insufficient.

10. Water saturation may be too high if samples contain large amounts of gypsum (see 4.8) or montmorillonite clays (water of hydration). Permeability and porosity values can also be altered if waters of hydration that are present in the reservoir are removed during extraction and drying procedures (humidity oven drying may be preferred).

11. If the true oil density is not known, error is introduced into the calculation of oil saturation because an oil density value must be assumed.

b. Oil volumes are not found directly and may be inaccurate due to the following:

1. Additional water collected or lost from the sample as mentioned above.

2. Loss of solids.

3. Incomplete cleaning of oil.

4. Drying at a higher temperature than the extraction temperature may remove additional water of hydration and overstate the oil volume.

c. Rock wettability may be altered.

d. The clay fabric may be altered, that may result in inaccurate permeability measurements.

e. There is no check on the analysis accuracy.

4.3.1.7 Accuracy/Precision

There are no documented standards and the accuracy of the methods cannot be assessed. However, with the calibration procedures noted in 4.3.1.8, water volume reproducibility can be evaluated. Similar procedures assess oil volume accuracy. On relatively small samples or samples containing high gas saturation with residual volumes of oil and water, the percent error for liquid saturations may be ±50 percent of values measured. Percent error will be significantly less as liquid volumes increase.

4.3.1.8 Calibration

The water measurement accuracy should be checked regularly to be sure no bias affects the results.

a. For gravimetric determination—Known weights of water added to the extractors are plotted against the weights of water recovered by extraction under conditions identical to those used in extracting an oil sand sample. The water correction factors may change due to the efficiency of the condensers used in the apparatus. Typical values are listed below:

$$\text{Corrected Weight Water} = (\text{Weight Water} \times a) + b$$

Where: a and b, respectively, are the slope and intercept of the calibration equation, that is, $a = 1.003, \pm 0.001$; $b = 0.090, \pm 0.009$.

b. For volumetric determination—A calibrated buret is used to deliver known volumes of water into the water trap. The traps should have a maximum scale error of 0.02 ml. Volume correction factors can be calculated and applied, if necessary.

4.3.2 Full Diameter Cores

4.3.2.1 Principles of Analysis

The distillation extraction method of determining fluid saturations for full diameter samples is governed by the same principles and procedures as those for plug samples (see 4.3.1.1). Differences in the procedure and apparatus are discussed below.

4.3.2.2 Apparatus

The following apparatus are appropriate for this method:

a. Flask—The flask used to accommodate the full diameter core sample must be larger than the plug analysis flask with a larger solvent volume for oil removal.

b. Trap—The trap or sidearm has to be large enough to accommodate the volumes of water from large samples. The graduated section of the trap should be marked in 0.1 ml divisions.

c. Extraction core protector—Extraction thimbles are not generally used for full diameter samples. Instead a stocking material made from unbleached cotton can be used to prevent the loss of fines from the full diameter core. Thimbles normally used in plug core analysis are not manufactured in sizes that fit the full diameter core.

4.3.2.3 Procedures

The procedures are the same as those for plug samples with the exception that sample weights should be made to the nearest 0.1 gram. Also, the cleaning time of 48 hours may have to be extended in order to fully clean the larger volume samples. Longer times and supplemental cleaning are frequently required.

4.3.2.4 Calculations

See equations in 4.3.1.4.

4.3.2.5 Advantages

Refer to 4.3.1.5. There are additional advantages when performing this test on full diameter cores, as follows:

a. A high precision analytical balance is not required as the weights need only to be determined to the nearest 0.1 gram.

b. Grain loss during the handling and testing procedures is not as critical as with plugs, but still should be minimized.

c. Collected water volumes are quite large as compared to those associated with plug analyses, therefore the water readings need to be recorded only to the nearest 0.1 ml.

d. As the oil volumes are determined by gravimetric difference the final values are not impacted to the same degree by small measurement errors as those determined on plugs, except at very low oil saturations.

4.3.2.6 Limitations

There are additional limitations when analyzing full diameter cores as follows:

a. Larger, more costly equipment is required, e.g., glassware, ovens, cut-off saws.

b. Laboratory space requirements are greater.

c. Large volumes of solvents are used thereby requiring a higher investment in same. Storage and/or disposal logistics are more complex.

d. Longer distillation and core cleaning times are required thereby decreasing data reporting timeliness.

e. Health and safety standards should be carefully monitored and observed due to the large volumes of fumes and solvents that can be involved.

4.3.2.7 Accuracy/Precision

Refer to 4.3.1.7.

4.3.2.8 Calibration

Refer to 4.3.1.8.

4.3.3 Pressure-Retained Core Analysis

4.3.3.1 Introduction

The objective of pressure-retained core analysis is to provide fluid saturation data on cores for which fluid expulsion has been minimized during core barrel retrieval by preventing pressure depletion from bottom hole to surface conditions. A ball valve seals the core barrel and prevents the natural pressure drop as the barrel is brought to the surface. Additionally, the barrel is designed to allow preset pressure maintenance during retrieval with a pressure supply that compensates for pressure loss due to a lower surface temperature.

Flushing of the core with drilling fluid filtrate before the core enters the core barrel is minimized with special, low-water-loss drilling fluid, a high coring penetration rate, a low drilling fluid overbalance, and a special core bit design. In addition, prior to coring, the inner barrel is generally filled with a low-invasion gel material. This material minimizes further drilling fluid filtrate invasion via imbibition by displacing drilling fluid from the core surface as the core enters the barrel.

Fluid saturations are the amounts of oil and water present in the core at laboratory conditions. Alteration of original fluid saturations by flushing can only be qualitatively defined. Any drilling fluid filtrate invasion alters saturations from in situ values unless the in situ saturations are at waterflood residuals. In under-pressured reservoirs, filtrate invasion is minimized by using foam as the drilling fluid. With the foregoing precautions, saturations under these conditions can reflect saturations near in situ values.

Rig site handling procedures are addressed in 2.2.5.

4.3.3.2 Principles of Analysis

The cores are kept frozen with dry ice until the laboratory analysis is initiated. This reduces pore pressure, freezes water, immobilizes oil, and traps gas that does not freeze or liquefy at dry ice temperature.

As the core is allowed to thaw in the gas collection cells, gas comes out of solution and expels both oil and water that are captured in a receiving tube at the bottom the cell. Gas is collected in the void space within the cell. This fluid volume does not necessarily reflect the amount of fluid produced by pressure depletion of a conventional core since both different pressure and different temperature conditions exist in the reservoir.

Since oil volumes are determined from the distillation extraction and gas driven solvent extraction steps of the analysis, grain loss must be kept to a minimum for maximum accuracy.

If no free gas is present in the cored zone and the core is captured above the bubble point of the oil, the total liquid saturation should be equal to the pore space after a formation volume factor is applied to the oil. Saturations should not vary more than ±5 percent.

Filtrate invasion analysis allows assessment of the fluid saturation alteration from in situ values. If available, relative permeability fractional flow curves will aid in this assessment. Detailed discussion of invasion principles is provided in 4.3.7.

4.3.3.3 Apparatus

The following equipment is necessary for the analysis of pressure-retained core. These items are considered exclusive to the analysis of pressure-retained cores. Standard core analysis items used in the analysis are not included.

a. Core storage boxes—Boxes used for storage of the frozen core until analysis is initiated. The boxes should be of sufficient size to hold 5-foot (1.52-meter) sections of core in its inner barrel and enough dry ice to maintain the core in a frozen state. These boxes should be insulated to minimize dry ice sublimation.

b. Dry ice—Used to maintain the core in a frozen state until analysis is initiated.

c. Liquid nitrogen—Used for milling of core barrels and during sample preparation such as core cleaning, drilling plugs, and full diameter sample facing.

d. Milling machine—Used to cut diametrically opposed grooves down the length of the inner barrel to facilitate frozen core removal.

e. Two-liter dewar flask—Used for periodic dipping of samples into liquid nitrogen during the cleaning process to aid in cleaning and to keep the core frozen.

f. Drilling fluid or low invasion gel removal tools—Various tools including carpet knife, insulated gloves, small hammer, etc.

g. Gas collection cells—Cell equipped with a pressure gauge to determine gas volumes bled from the core and core barrel, and to allow for chromatographic gas analysis.

h. Gas collection cylinders (250 cm^3)—Used for sampling of cell gas for chromatographic analysis.

i. Centrifuge—Used for centrifuging gas collection cell receiving tubes to obtain good oil/water/solid separation.

j. Heat lamp—Used for gas mixing in the gas collection cell prior to sampling.

k. Portable vacuum pump—Used for pulling a vacuum on the gas collection cell and 250 cm^3 cylinders prior to gas collection.

l. Stainless steel tubes [3-inch (76.2-millimeter) diameter with screen bottoms]—The core samples are placed in these tubes, thereby preventing grain loss from samples during the gas collection and distillation extraction steps of analysis.

m. Surgical stocking material—Used to encapsulate core samples during CO^2 extraction process to minimize grain loss.

4.3.3.4 Procedures

4.3.3.4.1 Core Preparation Procedures

Core preparation procedures include:

a. Core samples, encased in steel tubing and frozen in chests of dry ice, are brought from the wellsite to the laboratory.

b. Each length of tubing-encased frozen core is placed into a dry ice filled trough attached to a milling machine. Two diametrically opposed grooves are milled down the length of the steel tubing to a depth slightly less than the wall thickness of the tubing. Liquid nitrogen is directed at the point of milling to ensure a tubing and core temperature at or below the dry ice temperature.

c. The tubing is separated into two halves and the frozen core is removed.

d. The low invasion gel that is frozen on the core is removed by scraping and brushing. The core sections are periodically dipped in liquid nitrogen to ensure that the core remains frozen.

e. Cores are visually examined for lithological characteristics and samples are selected for analysis.

f. The ends of each selected core segment are faced with a diamond saw using liquid nitrogen as a coolant. The faced frozen core segments are wrapped in plastic wrap and aluminum foil and stored under dry ice to await laboratory tests. The plastic wrap and foil protect the core from sublimation damage.

g. Care while milling open the core barrel ensures safe core handling. Placing a heavy mesh net over the core barrel during the milling process prevents analyst injury should the core barrel separate due to excessive internal pressure caused by drilling fluid expansion during freezing. Natural gas pockets where core is missing in the barrel may ignite in air. The carbon dioxide and nitrogen environment surrounding the core barrel during milling should prevent gas ignition.

4.3.3.4.2 Gas Collection Procedures

Gas collections procedures include:

a. The faced frozen core sample is placed in a thin-walled, metal cylinder with a fine mesh screen in the bottom, weighed quickly, and placed in the gas collection system (see Figure 4-6).

b. The system is immediately assembled and evacuated for 45 seconds to remove air without pulling gas from the sample. The frozen core is then allowed to thaw to room temperature.

c. Water and oil expelled by the evolving gas are collected in a graduated receiving tube attached to the gas collection cell.

d. The evolved gas is collected in the gas collection cell. The system is equipped with a pressure gauge to allow pressure monitoring inside the cell. If the gas collection cell pressure exceeds 0 psig, an attached and previously evacuated secondary cell is then opened to allow collection of additional evolved gas.

e. Barometric pressure, room temperature, system pressure, and produced liquid volumes are recorded periodically. Thawing of the core is considered to be completed when consecutive readings indicate system equilibrium.

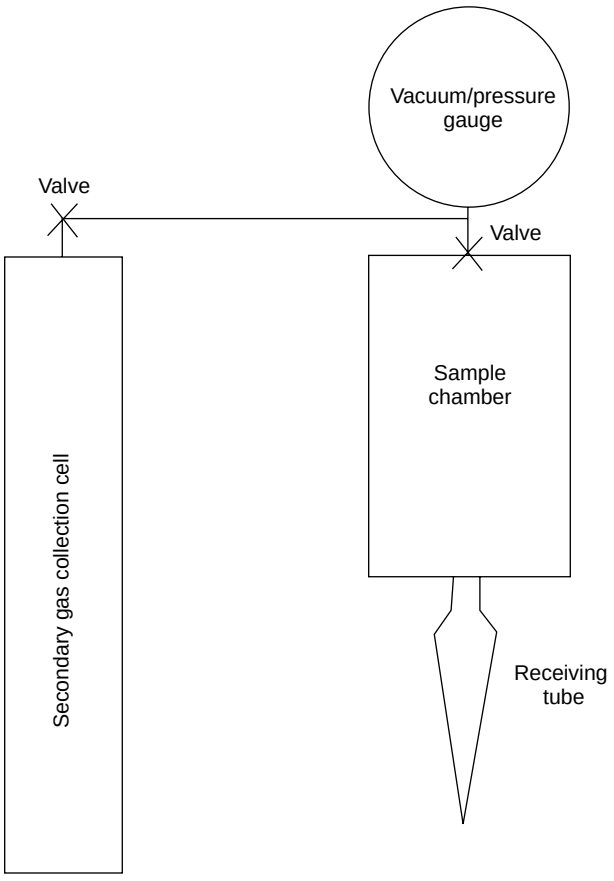

Figure 4-6—Gas Collection/Pressure Depletion System

f. Evolved gas samples may be collected in more than one cell. Gas samples are collected separately from the primary collection cell and, if used, the secondary gas collection cell. The gas samples are analyzed to determine gas gravity and component mole percent. Volumes of oil and water collected are measured. The chloride and bromide content of the water and the produced oil gravity are determined.

g. The sample and its cylinder are removed from the cell, weighed, and placed in a previously prepared distillation extraction apparatus.

4.3.3.4.3 Distillation Extraction (Dean-Stark) Procedures

Distillation extraction procedures include:

a. The sample is placed in the distillation extraction apparatus (see 4.3.1.2), the system is assembled, and heat is applied to distill the remaining water in the core and to extract the remaining oil.

b. When the distillation is complete, as determined by consecutive water readings, the water volume is recorded. The sample and thimble are then removed and placed in a vacuum oven at 240°F (116°C) to remove the extraction solvent. When drying is completed, the sample and thimble are removed from the oven, allowed to cool in the presence of a desiccant, and then reweighed.

c. The volume of additional oil extracted is determined gravimetrically, using an oil density determined from the oil produced in the gas collection step. The volume of water distilled is corrected to reflect the equivalent volume of water having the same salinity as the water recovered during the gas collection phase. If the water expelled during the gas collection is contaminated with drilling fluid filtrate, the water recovered during the gas collection phase may not have the same salinity as less contaminated formation water at the center of the core.

d. The core sample with all loose grains is then encased in a surgical stocking material to minimize grain loss prior to additional extraction procedures.

4.3.3.4.4 Gas-Driven Solvent Extraction and Saturation

Gas-driven solvent extraction and saturation procedures include:

a. The cores are weighed in the stocking material and placed in the gas-driven solvent extractor where they are subjected to further cleaning using carbon dioxide-charged toluene heated to 180°F (82°C). This phase is designed to remove any oil remaining. The weight loss resulting from this extraction process is the weight of additional oil removed that is converted into an oil volume using the previously calculated oil density. In actual practice, the total weight loss from distillation

extraction and gas-driven solvent extraction minus the weight of water recovered is used to determine oil volume.

b. The samples are then placed into a convection oven (or vacuum oven) and dried at 240°F (116°C) until weights stabilize. For samples containing large amounts of montmorillonite and other clays, a humidity oven can be used to help preserve a single or double water layer to better approximate expected clay/water interactions in the reservoir. (Humidity levels during drying are important in maintaining proper waters of hydration.)

c. Full diameter porosities along with horizontal air permeabilities are measured on each core segment. In the cases where more than one sample is combined in a foot of analysis, these values are weighted by representative portions of a foot and averaged to arrive at a mean value.

d. Liquid saturations at stock tank conditions are calculated using the measured total pore volume of all of the core segments comprising the sample and the total oil and water contents recovered from these segments. The volume of gas collected from the samples is then determined from the known volume of the gas collection system, corrected for the grain and tare volumes as well as the total liquid and salt contents. This gas volume is then further corrected to standard conditions.

4.3.3.5 Calculations

The following calculations are used for this method:

a. Gas volume at standard temperature and pressure and gas weight from gas collection:

$$\text{Gas Volume} = \frac{\text{Gas Collection Cell Volume} \times \text{Cell Pressure} \times 520 \times (1 - \text{Air Fraction})}{14.7 \times (460 + \text{Temperature})}$$

$$\text{Gas Weight} = \text{Gas Volume} \times 0.0012232 \times \text{Gas Gravity}$$

Where:

Gas collection cell volume is in ml.
Cell pressure is in psia.
Temperature is in degrees F.
Gas weight is in g.
Gas volume is in ml at STP.
Gas gravity = (density gas, g/ml)/(density of air, g/ml).
Density of air at STP = 0.0012232g/ml.
STP is standard temperature (0°C) and pressure (1 atmosphere).

The (1 – air fraction) term is a correction factor. Evacuation of the gas collection cells removes all but about 5 percent of the air in the system and small leaks may develop at the seal lid allowing air to be pulled into the system. Analysis of a gas sample drawn from the collection cell determines the amount of air present.

The gas weight is used in checking the material balance of the total weight loss as a result of extracting gas, oil, and water from the sample with the calculated weight loss.

b. Saturations:

$$\text{Oil Saturation} = \frac{(\text{gcc Oil Vol} + \text{Dist Ext Oil Vol} + \text{CO2 Ext Oil Vol}) \times 100}{\text{Pore Volume}}$$

$$\text{Water Saturation} = \frac{(\text{gcc Water Vol} + \text{Dist Ext Water Vol}) \times 100}{\text{Pore Volume}}$$

Where:

gcc Water Vol = gas collection water volume, cm^3.
gcc Oil Vol = gas collection oil volume, cm^3.
Oil saturation is given as percent pore volume.
Water saturation is given as percent pore volume.
All oil and water volumes are in ml.

c. Water volume from distillation extraction—See 4.3.2.

d. Pore volume and grain density correction—When salts are not leached from the core samples, corrections in pore volume and grain density are made based on water salinity determined from the water produced during gas collection and pressure depletion. Alternatively, methanol can be used to leach salt from the core.

$$\text{Pore Volume (corrected)} = \text{Pore Volume} + \frac{\text{Water Volume} \times \text{Salinity NaCl}}{C}$$

Where:

Pore volume is in ml.
Water volume from distillation extraction is in ml.
Salinity NaCl is in grams per ml of distilled water.
C = 2.165 g/ml (density of NaCl).

$$\text{Grain Density (corrected)} = \frac{\text{Sample Weight} - (\text{Water Volume} \times \text{Salinity NaCl})}{\text{Bulk Volume} - \text{Pore Volume (corrected)}}$$

Where:

Grain density is in g/ml.
Water volume from distillation extraction is in ml.
Salinity NaCl is in g/ml.
Bulk volume is in ml.
Pore volume is in ml.

4.3.3.6 Advantages/Limitations

Advantages/limitations of this method are:

a. Pressure-retained coring is an expensive, meticulous, and time-consuming operation. It requires a straight and clean hole containing a specially formulated drilling fluid or foam.

b. A laboratory including the necessary special equipment and experienced supervisors should be located as close as

possible to the well location since it may be difficult to safely transport a frozen core over great distances.

c. Pressure-retained core analysis provides fluid saturations more indicative of in situ saturation values than those values obtained by conventional core analysis. The ability to prevent fluid expulsion during core retrieval (pressure depletion) prevents saturation alteration during core retrieval. Refer to advantages/limitations for distillation extraction cleaning (see 4.3.1.5 and 4.3.1.6) to understand the impact on saturation determination.

d. Minimizing pore space flushing during coring is another factor that must be considered in order to obtain near in situ saturations. Pressure-retained coring procedures are designed to prevent alteration from depletion, but not from flushing. As a result, core saturations, from cores normally recovered in water base drilling fluids, are indicative of in situ oil saturation values only if filtrate flushing is minimal (see 4.3.7). If a large amount of flushing occurs, then the measured saturations are more indicative of residual values. This is important for the evaluation of tertiary recovery projects.

e. Flushing is minimized by minimizing drilling fluid overbalance during coring, through use of low fluid loss drilling fluid, and by penetrating the formation as rapidly as possible while maintaining good core recovery. In certain situations, i.e., depleted bottom-hole pressures, a stabilized-foam drilling fluid minimizes flushing. However, if the foam breaks down during coring, the core is subjected to a high surfactant fluid loss and appreciable flushing occurs.

f. The ability to capture oil and water in the core provides a distinct advantage over conventional and sponge core analysis. By capturing all water that existed in the core at the completion of coring, it is possible to determine the filtrate volume that displaced gas, oil, or water during coring operations. By knowing the measured saturations and the filtrate amount, saturation alteration during coring is qualitatively assessed by using the formation relative permeability fractional flow characteristics.

In comparison, conventional and sponge cores allow determination of only the residual core filtrate after coring because fluids are expelled from the core during retrieval. However, since most filtrate is located in the core's periphery, the measurement of filtrate is erroneously low. In a conventional core, the oil saturation is also reduced during pressure depletion, although this oil may be retained by the sponge in a sponge core and analyzed. If oil saturations are near residual, the k_g/k_o is such that usually very little oil is expelled by gas drive.

g. Since the gas is retained in the pressure-retained core, it is possible to measure gas volume as well as gas composition from a pressure-retained core. This is important when evaluating sweep efficiencies from gas injection. Residual saturations in highly-swept zones allow assessment of various petrophysical rock properties at residual saturations after gas injection.

h. Gas-oil ratios, when averaged over an interval, have agreed favorably with production measurements.

i. Oil gravity can be determined on a foot-by-foot basis throughout the cored interval to determine if oil gravity changes with depth. Gravity changes must be appreciable to be observed by this method.

j. The diameter of pressure cores is presently 2.5 to 3.75 inches (64.5 to 95.3 millimeters). The smaller diameter core increases the surface area to volume ratio of the sample. The effect of drilling fluid filtrate invasion and saturation alteration is greater than in larger-diameter core. As in any core analysis measurement, the degree of accuracy is decreased as sample size is reduced; therefore small measurement errors usually will have less impact on pressure-retained core analyses as the core diameter is increased.

4.3.3.7 Accuracy/Precision

Accuracy/precision of this method is:

a. Fluid saturations—Accuracy limits of fluid determination from each phase of analysis are as follows:

1. Gas collection: fluid volumes are ±0.5 ml; water volumes (distillation extraction) are ±0.5 ml.
2. Gas driven solvent extraction: oil volume is ±0.1 g or ±0.1 ml.

b. Gas volume—Gas volume is determined by Boyle's Law. Resulting gas volume is ±2% of actual volume. The variables in calculation of gas volumes have an accuracy as follows:

Cell volume	=	±5 ml.
Cell temperature	=	±0.5°F.
Cell pressure	=	±0.2 psia.
Air fraction	=	±0.5 mol %.

4.3.3.8 Calibration

Calibration issues for this method include:

a. Receiving tubes on gas collection cells and distillation extraction apparatus should be calibrated using deionized water on a scale reading to 0.01 grams. Weights at each interval are converted to volumes using the water density at the appropriate temperature. Volumes should be determined for each 10 ml reading on the receiving tubes. Interpolation is used to determine true volumes between the calibrated volumes.

b. Gas collection cell volumes should be calibrated using an accurate gasometer or meter. Each cell is evacuated and the volume determined. Repeatability of measured volumes shall be within 5 ml.

c. Balances should be checked at regular intervals for accuracy.

4.3.4 Sponge Core Analysis

4.3.4.1 Principles of Analysis

In sponge coring, the core enters a half-inch thick polyurethane or cellulose/polyurethane sponge sleeve inside an aluminum liner. As the core is brought to the surface, expanding gases displace crude oil that is captured by an oil-wet sponge sleeve (or water that is captured by a water-wet sponge sleeve).

The oil-wet sponge sleeve consists of polyurethane sponge with about 70 percent porosity that is easily compressed to almost no porosity. The sponge is saturated with brine prior to going into the well. If the formation brine properties are known, the brine used to saturate the sponge should match the salinity and density of the water in the formation to be cored. Any oil that comes out of the core will displace water in the sponge and will spread out in a thin layer over the walls of the sponge pores.

The water-wet sponge consists of a cellulose fiber mixed in with the polyurethane. The sponge is saturated with dry mineral oil prior to going into a well cored with an oil-based drilling fluid. The objective is to capture any water escaping the core as it is brought to the surface.

4.3.4.2 Apparatus

4.3.4.2.1 Equipment

High speed table saw with at least a 10-inch (25.4-centimeter) diameter carbide blade and 1.5 horsepower motor to open the aluminum core barrel.

4.3.4.2.1.1 Oil-Wet Sponge Analysis Equipment

The following additional equipment is required for oil-wet sponge analysis.

a. Container to hold sponge (large Soxhlet, metal can, etc.).
b. Spectrometer for determining amount of crude in solution, e.g.:
   1. Visible spectrometer (detects intensity of crude color).
   2. Ultraviolet-fluorescence spectrometer (detects aromatics).
   3. Near-infrared (NIR) spectrometer (detects C-H bonds, asphaltenes).
   4. Nuclear magnetic resonance (NMR) spectrometer (detects the C-H shift).
c. Analytical balance for preparing crude extract standards used in calibrating spectroscopic response.
d. Gas chromatograph (optional).

4.3.4.2.1.2 Water-Wet Sponge Analysis

In addition to 4.3.4.2.l, water-wet sponge analysis requires a standard distillation extraction apparatus (e.g., Dean-Stark) for determining water volume recovered (see 4.3.2).

4.3.4.2.2 Reagents

4.3.4.2.2.1 Oil-Wet Sponge Analysis

Any good crude-oil solvent that has:

a. No visible color, if using visible spectrometer.
b. No aromatics, if using UV-fluorescence spectrometer.
c. Preferably no C-H bonds, if using NIR or NMR (e.g., hydrogen-free chlorofluorocarbons, or chlorocarbons).

4.3.4.2.2.2 Water-Wet Sponge Analysis

Any standard distillation extraction solvent, e.g., toluene.

4.3.4.3 Procedure/Precautions

4.3.4.3.1 Rig Site Handling

At the rig site, care should be taken not to jar the ends of the sponge core liner, as this can cause the rock core to shift in the barrel resulting in a mismatch of the sponge and rock core. Although the sponge core should be kept cool, freezing it with the rock core still inside carries some risk of inducing fractures in the rock core.

For certain formations, such as fractured or vuggy carbonates, core can break and wedge into the sponge causing it to bunch together. The larger-diameter sponge core is less susceptible to this problem.

Immediately after retrieval, the cores may be cut into sections (typically about 5 feet) and stored in transport containers filled with the same fluid used to saturate the sponge.

4.3.4.3.2 Oil-Wet Sponge Analysis

Because the sponge is presaturated with water and is highly compressible, any analysis of the amount of oil in the sponge should measure oil directly, and not infer oil volume from a measurement of the water in the sponge and the porosity (as is done in the distillation extraction method for measuring oil saturations of rocks).

Because the oil in the sponge is spread out in a thin layer over the oil-wet sponge, mechanical compression of the sponge is not a good method for trying to recover the captured oil.

A large-bore Nuclear Magnetic Resonance instrument, such as an NMR imager, can be used to directly determine the amount of oil in the sponge, provided the sponge sleeve is removed from the electrically-conducting aluminum liner. The direct NMR method will become more attractive as the measurement cost declines and NMR availability increases.

As of 1993, the least expensive, most widely applicable method, and the method recommended here, is solvent dissolution of the captured crude followed by a spectroscopic determination of the amount of crude in solution. Since sponge may be subject to attack by solvents during the solvent dissolution step, one must either select solvents that are gentle

on sponge or be sure that the subsequent analysis is unaffected by dissolved sponge components mixed in with the dissolved crude. For spectroscopic determination, a calibration curve should be developed by injecting known weights or volumes of lease crude into pieces of sponge, then solvent-extracting the sponge and determining the spectroscopic response of the extracts. Sponge samples should be taken from the actual lot of sponge used in the sponge coring operation. If different lots of sponge are used during coring, a new calibration curve should be developed for each new sponge lot.

4.3.4.3.3 Water-Wet Sponge Analysis

The water-wet sponge is hygroscopic, so care must be taken to prevent absorption of moisture from the atmosphere because this could impact the final analysis for the water saturation. Standard distillation extraction analysis can be performed on water-wet sponge to determine water content.

4.3.4.3.4 Separation of Sponge Sleeve and Aluminum Liner From Rock Core

With a table saw, make two cuts 180 degrees apart down the length (long axis) of the aluminum liner that jackets the sponge sleeve. Set the depth of cut to 0.25 inches (6.35 millimeters) so that the saw blade cuts only through the aluminum liner and not the sponge sleeve. Also, make the cut between a pair of aluminum centralizer vanes (the vanes that hold the rock core centered within the aluminum liner) instead of on top of a vane.

Next, cut through the sponge sleeve with a knife and remove each section of whole core from the liner for trimming. Note any evidence that the core has shifted relative to the liner. Mark the liner so it can be cut to the same length as the corresponding section of trimmed core and label the matching pieces of whole core and liner.

Complete the process of cutting and sealing the rock and sponge samples within 30 minutes to minimize exposure to the atmosphere. This minimizes evaporation of core fluids, oxidation of liquid crude, and the resulting errors in quantifying the amount of crude. If the sponge is not immediately analyzed, carefully preserve it until it is analyzed (see 2.2.6 for preservation methods).

The corrected whole-core oil (or water) saturation is obtained by adding the volume of oil (or water) in each piece of trimmed core to the volume of oil (or water) in the matching piece of sponge sleeve and dividing by the total whole-core pore volume.

4.3.4.3.5 Solvent Extraction and Spectroscopic Analysis (Oil-Wet Sponge)

Analysis of oil-wet sponge is as follows:

a. Prepare sponge samples with known amounts of lease crude and extract sponge with chosen solvent. Calibrate the spectroscopic response using these extracts.

b. Place sponge core crude into solution by any of the following:

1. Soxhlet extraction.
2. Submerged soak and vigorous shaking.
3. Submerged soak and disaggregation of sponge in blender.

For options (2) and (3) it will be necessary to separate the sponge sleeve from the aluminum liner.

4.3.4.4 Calculations

4.3.4.4.1 Oil-Wet Sponge Analysis

Calculations for oil-wet sponge analysis are as follows:

a. Determine the spectroscopic response of an aliquot of the solvent extract.
b. Knowing the total amount of solvent extract and using a calibration performed with prepared standards, determine the total amount of crude that was originally imbibed by sponge.

$$V_{tc} = W_{tc} / \rho_c = (R_a / R_{pwf}) (W_{ts} / \rho_c)$$

Where:

R_{pwf} = Response of spectrometer per weight fraction of sponge-imbibed lease crude in the final solvent extract. This response calibration is done using prepared standards consisting of solvent extracts of sponge samples (of the current lot) that have been injected with known amounts of lease crude. (For example, absorbance or fluorescence of solvent extract per weight fraction of sponge-imbibed crude when using a given sample cell at a given wavelength of light.) Units of R_{pwf} must be the same as the units of R_a.

R_a = Response of spectrometer for an aliquot of the solvent extract recovered from a piece of sponge. Response is obtained under the same conditions (e.g., using same sample cell, wavelength, etc.) that were used when calibrating the spectrometer response with prepared standards. Units of R_a must be the same as the units of R_{pwf}.

W_{ts} = Weight of the total crude solution recovered from a piece of sponge core. Weight is measured before the small aliquot is removed, g.

W_{tc} = Total weight of crude that was in a piece of sponge core, g.

V_{tc} = Total volume of crude that was in a piece of sponge core, cm^3.

ρ_c = Density of lease crude, g/cm^3.

4.3.4.4.2 Water-Wet Sponge Analysis

Knowing the brine salinity and density, the volume of brine that was in the sponge can be calculated from the volume of distilled water recovered from the sponge as follows:

$$V_{br} = [(V_w)(\rho_w)/\rho_b]\ [1{,}000{,}000/(1{,}000{,}000 - C_s)]$$

Where:

V_{br} = Volume of brine corresponding to the volume of distilled water collected from a piece of sponge, cm^3.
V_w = Volume of distilled water collected from a piece of sponge (e.g., Dean-Stark), cm^3.
ρ_w = Density of distilled water, g/cm^3.
ρ_b = Density of lease brine having a concentration, C_s, of salt, g/cm^3.
C_s = Concentration of dissolved salts in lease brine = (1,000,000) (weight of salt)/(weight of brine), ppm.

4.3.4.5 Advantages/Limitations

Sponge core is a less expensive alternative to pressure-retained coring (as described in 4.3.3) and is operationally simpler. The amount of oil (or water) captured in the sponge is added to the amount of oil (or water) remaining in the core to obtain more accurate values of oil (or water) saturation.

Unlike pressure core, any hydrocarbon gases that are released are not retained, and so are not available for analysis. Also, with sponge core, a choice must be made between determining corrected values of the core's oil saturation or determining corrected values of the core's water saturation. Both corrected saturations cannot be determined using the same sponge core.

4.3.4.6 Accuracy/Precision

For extraction and spectroscopic analysis combined, the oil volume from oil-wet sponge core analysis is within 5 percent of the actual volume of oil or 0.2 ml of oil, whichever is larger. The water volume from water-wet sponge core analysis is within 0.1 ml of the actual volume of water from which the corresponding volume of brine can be calculated if the brine salinity and density are known. The water volume is determined more accurately than the oil volume because the water is determined by a direct measurement of volume. In contrast, the oil sample is highly diluted in solvent and only an aliquot of the dilute solution is analyzed spectroscopically.

4.3.4.7 Calibration

Refer to 4.3.1.8. Standard calibration procedures should be followed for all commercial equipment.

4.3.5 Productive Shale

4.3.5.1 Principles of Analysis

This paragraph covers the special considerations for measuring fluid saturations of the pore space in "oil productive shale"—rocks composed of clay-to-silt-sized particles and potentially oil productive. Analysis of samples from this type formation may need special handling due to their low permeability, possible presence of tightly bound or structural water, and potential for solid organics. A retort process is not recommended for samples containing solid organics. Oil will be generated from the solid organics at temperatures used in this process yielding high oil volumes. A distillation process should be used to determine fluid saturation data.

The principle is the same as in 4.3. This method should be used if solid organics present are not to be included as oil volume. If shorter analysis times are needed, removal of hydrocarbons can be expedited by crushing the sample. The crushed sample will also expedite equilibrium time for grain volume measurements.

4.3.5.2 Apparatus

The apparatus for distillation extraction is the same as in 4.3.1.2. For sample-crushing, a mortar and pestle are needed.

4.3.5.3 Procedures/Precautions

The procedures are the same as in 4.3.1.3. If samples are to be crushed to expedite hydrocarbon removal or grain volume determinations, then bulk volumes should be measured and samples weighed prior to crushing. Crush sample using mortar and pestle and reweigh. If there is weight loss during crushing, a correction to either rock or fluid volumes must be made. If weight loss is high, the sample should be discarded and another sample tested. Careful control of sample loss throughout the procedure is critical to accurate oil volume measurement.

4.3.5.4 Calculations

Calculations should be performed as stipulated in 4.3.1.4.

4.3.5.5 Advantages

Advantages of this method include:

a. All measurements can be made on the same sample.
b. Solid organics are not represented in oil volumes.
c. Accurate grain densities can be measured.

4.3.5.6 Limitations

Limitations of this method include:

a. Oil volumes are sensitive to drying techniques.
b. Long extraction times are needed due to the low permeability of this type of rock material.
c. Sample loss is critical in determination of oil content.

4.3.5.7 Accuracy/Precision

Proper distillation and drying techniques should permit oil and water content determination to within 2 percent of the values measured. Special care is necessary when drying samples because of the rocks' ability to rehydrate quickly, which will cause erroneously low oil volumes.

4.3.5.8 Calibration

Calibration techniques should be performed as outlined in 4.3.1.8.

4.3.6 Tar (Oil) Sand Analysis

4.3.6.1 Principles of Analysis

The distillation extraction method can be used to determine the fluid saturations of unconsolidated tar (oil) sands. However, certain modifications to the procedure and data presentation are required. The required changes are described in the following paragraphs.

4.3.6.2 Apparatus

The same basic equipment as described in 4.3.1.2 may be used in this procedure.

4.3.6.3 Procedures/Precautions

The unconsolidated core may be frozen prior to obtaining a sample. The plug should be drilled using liquid nitrogen as a lubricant. The frozen plug is then sleeved, usually with heat shrinkable polytetraflouroethylene (PTFE), in order to keep the plug intact when the bitumen is removed. The sample is then placed in the distillation extraction apparatus (see Figures 4-4 and 4-5) with toluene as the solvent. In some instances, the core may not be frozen, or if the sample is obtained during the quarry process of a tar sand deposit, the test sample may be analyzed on a volume/weight basis. In such instances, the test specimen is transferred to an appropriate extraction thimble such as those discussed in 4.3.1.2.(f).

Avoid violent boiling by supplying a minimum heat rate to boil the solvent. Change the toluene, as required, to prevent tar build up and subsequent "bumping" or super heating. This latter condition, should it occur, can slow down the distillation/extraction process or possibly negate the complete test.

If the bitumen has an initial boiling point greater than 392°F (200°C), the oil content can be determined directly. For direct determination, the toluene-bitumen bottoms are transferred into a volumetric flask and toluene is added until the total volume equals the volumetric flask's volume. An aliquot is taken from the flask and placed on glass fiber filter paper. The toluene is evaporated and the bitumen is weighed. The total bitumen weight is then calculated. This method is known as the Modified Dean-Stark (distillation extraction) Method or AOSI-3573.[4]

4.3.6.4 Calculations

The liquid contents of the sample are usually reported as weight percent as opposed to percent pore volume.

$$\text{Water Content} = [(V_w \times \rho_w)/\text{Sample Weight}] \times 100$$

$$\text{Oil Content} = \frac{[(\text{Sample Weight}) - (V_w \times \rho_w) - \text{Dry Weight}] \times 100}{\text{Sample Weight}}$$

Where:

Water Content = weight percent of the sample weight.
Oil Content = weight percent of the sample weight.
V_w = volume of water recovered, ml.
ρ_w = density of water, g/cc.
Sample Weight = original (net) sample weight, gm; i.e., excludes sleeve weight.
Dry Weight of cleaned and dried sample, g.

Note: For practical purposes the density of the water may be assumed to be 1.00 g/cc.

(For ACOSA Method calculations, see 4.10, Reference 4.)

4.3.6.5 Advantages/Limitations

Advantages/limitations for this method include:

a. The liquid content values are determined on one sample.
b. The method is fairly rapid.
c. The calculations are not complex.
d. If the sample has a high tar (oil) content, it may be necessary to stop the distillation process so that the dirty toluene in the boiling flask can be replaced with clean toluene.

4.3.6.6 Accuracy/Precision

The weight percent values should be reported to the nearest 0.1 percent. Although there are no industry standards, the weight percent values should fall in the range of ±0.5 percent of the calculated value. This level of accuracy should be acceptable for most applications.

4.3.6.7 Calibration

Refer to 4.3.1.8.

4.3.7 Filtrate Invasion Analysis (Pressure Cores)

4.3.7.1 Principles of Analysis

The objective of filtrate invasion analysis is to quantify the amount of drilling fluid filtrate that invades the core during the coring operation. This is accomplished by adding a tracer(s) to the drilling fluid system in known quantities and measuring this tracer(s) content in the core. The degree of this tracer(s) concentration/activity/dilution allows determination of the drilling fluid filtrate amount contained in the core. Knowledge of the drilling fluid filtrate amount in the core allows a qualitative assessment of the saturation alteration that occurs by flushing during coring and calculation of the salinity of the formation water (see 7.7).

Filtrate invasion studies may be planned for other than pressure cores using a variety of water-based and oil-based coring fluids. Procedures for such studies will not be addressed in this publication. The wide range of core types

along with a similar broad spectrum of coring fluids and potential tracer materials precludes the recommendation of standard procedures for these other techniques.

Use of relative permeability fractional flow characteristics of the formation enhances the assessment of the fluid saturation alteration. The use of low-invasion profile coring bits minimizes drilling fluid filtrate invasion and saturation changes due to flushing.

The drilling fluid system is tagged with a tracer material. For water-base drilling fluid, tritiated water is recommended and is the basis of the following procedural descriptions. For oil base drilling fluid, tritiated hexadecane has been used.

The drilling fluid system is circulated sufficiently to allow even mixing of the tracer. A "rule of thumb" is to circulate the drilling fluid "bottoms up" three times. Periodically during coring, a drilling fluid sample is taken for use as a base reference for tracer concentration/activity in the drilling fluid for filtrate concentration calculations in the core.

A vertical plug is drilled through selected intervals resulting in "plug" and "donut" samples (see Figure 4-7). Water is extracted from these samples and a determination of the tritium activity is made by liquid scintillation analyses. The decrease in tracer concentration/activity in comparison with the drilling fluid filtrate is equal to the dilution of the drilling fluid filtrate by the formation water. The amount of drilling fluid filtrate invasion can be determined for both the exterior of the core and the interior of the core as well as the total drilling fluid filtrate invasion.

To minimize the extent to which drilling fluid filtrate migrates into the interior of the core by diffusion or imbibition, the samples should be cut at the well site as soon as possible after core retrieval. If this is not feasible, then the inner core barrel should be promptly frozen and preserved. Gidman and Conner, 1992[5] have reported that core freezing causes some fluid migration that may lead to incorrect assessment of the amount of drilling fluid filtrate invasion.

4.3.7.2 Apparatus

The following apparatus are used:

a. Drill press capable of cutting samples using liquid nitrogen as the bit lubricant.
b. Distillation extraction apparatus in 4.3.1.2 for plug analysis.
c. Drilling fluid filter press for extraction of the coring fluid filtrate.

4.3.7.3 Procedures/Precautions

Procedures and precautions include:

a. Use established safety procedures.
b. Designate selected intervals for the filtrate invasion study. Generally a sample is taken approximately every 4 feet (1.22 meters) throughout the cored section.
c. Care should be taken to avoid evaporation of water during handling of the core, plugs, and donuts.
d. The selected full diameter core intervals should be faced into right cylinders approximately 2 to 3 inches (50.8 to 76.2 millimeters) long. A vertical plug is drilled from the center of each sample to provide plug and donut portions of the sample (see Figure 4-7).

There are other methods to determine the spatial extent of drilling fluid filtrate invasion, such as cutting a plug perpendicular to the axis of the whole core. This plug is then sliced into many segments and each segment is analyzed for drilling fluid tracer.

e. The plug and donut samples are weighed and placed into separate Dean-Stark distillation extraction apparatuses to distill the water from the core and to extract the oil. Thimbles should be used to minimize grain loss. The water removed from each sample is measured, bottled, and marked. Since tritiated water has nearly the same chemical and physical properties as water, it is not separated from the ordinary water during the distillation process.
f. The samples are dried in a convection oven at 240°F (117°C) until weights are stable. The dry weight of each sample is recorded.

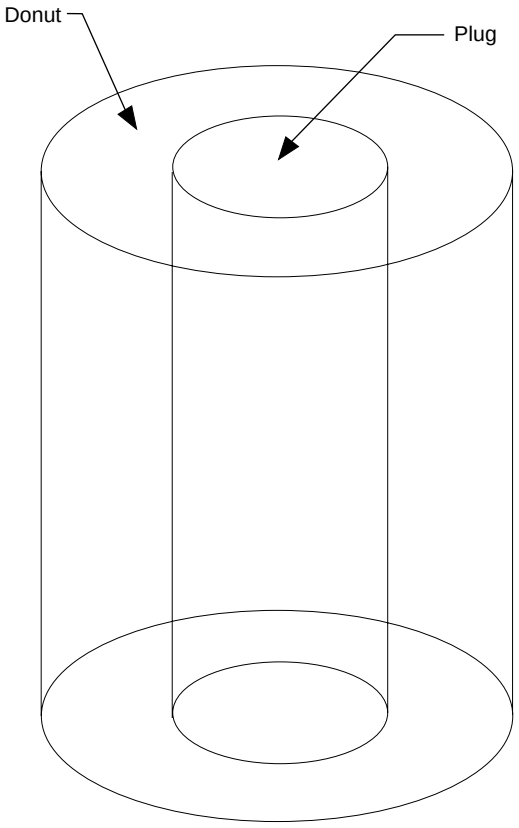

Figure 4-7—Plug and Donut Sample For Filtrate Invasion Study

g. Porosity is determined from measurements on the plug sample. The donut is assumed to have the same porosity as the corresponding plug. The pore volume of the companion donut is determined by the ratio of weight of the plug to the weight of the donut.

h. Water saturation is calculated for the plug and donut samples.

i. The amount of drilling fluid filtrate in the water extracted from each plug and donut sample pair is determined. The ratio of drilling fluid filtrate to total water in each sample is calculated and expressed as a percent of each sample's pore volume.

j. The analyst should observe appropriate safety measures when using tritiated water.

4.3.7.4 Calculations

The following calculations are appropriate for this method:

a. Water saturation in the plug and donut:

$$S_w\,(\text{plug}) = [S_{wt}\,(\text{plug})/P_v\,(\text{plug})] \times 100$$

$$S_w(\text{donut}) = [S_{wt}(\text{donut})/(P_v(\text{plug}) \times (\text{donut wt}/\text{plug wt})] \times 100$$

Where:

S_w (plug) = water saturation of the plug given as a percent of pore volume.

S_w (donut) = water saturation of the donut given as a percent of pore volume.

S_{wt} (plug) = total water volume of the plug, ml.

S_{wt} (donut) = total water volume of the donut, ml.

P_v (plug) = pore volume of the plug, ml.

The plug and donut weights are given in g.

b. Filtrate invasion in the plug and donut:

$$\text{Filtrate (drilling fluid)} = \frac{\text{Tritium Activity (sample)} \times 100}{\text{Tritium Activity (drilling fluid)}}$$

$$\text{Filtrate } (PV) = \text{Filtrate (drilling fluid)} \times S_w/100$$

Where:

Filtrate (drilling fluid) = drilling fluid filtrate as a percent of total water.

Filtrate (PV) = pore volume, percent.

S_w = water saturation, percent pore volume.

Tritium activity is in pCi/ml.

4.3.7.5 Advantages/Limitations

Advantages and limitation of this method include:

a. The advantage of determining coring fluid filtrate invasion on pressure-retained core as opposed to either conventional or sponge core is that all the water phase is retained within the core during retrieval since the core barrel pressure is maintained at original coring pressure. Thus, the quantity of drilling fluid filtrate contained in the core segments at the completion of the coring operations prior to surfacing can be quantitatively determined.

Fluids are expelled from conventional or sponge cores as the core barrel is retrieved from downhole to the surface. Since conventional cores contain a larger percent of drilling fluid filtrate in the periphery of the core, this filtrate is the first aqueous fluid to be expelled as the pressure drops during core barrel retrieval. The volume of drilling fluid filtrate is reduced by this fluid expulsion, and the amount of drilling fluid filtrate measured from these types of cores will be only the residual value.

Either a water-retaining or an oil-retaining sponge liner can be selected. The water expelled by gas evolution is recovered with a water-wet sponge and can be analyzed for drilling fluid filtrate tracer. The core fluids can also be analyzed for tracers by Dean-Stark extraction and analysis of the recovered waters.

b. In situ saturation may not be qualitatively determined even with knowledge of the amount of drilling fluid filtrate contained in the core segments. The drilling fluid filtrate displaces both oil and water. The amount of each phase displaced depends on the in situ saturations, fluid properties, and formation properties. If in situ, the formation is at irreducible water saturation, then the filtrate displaces mostly oil or gas. Conversely, if in situ, the formation is near residual oil saturation then mostly water or gas is displaced. Between these two extremes, oil and water displacement depend on the respective relative permeabilities. Relative permeability fractional flow curves should be used in assessment of the fluid saturation alteration during coring operations.

c. Tritiated water is the recommended drilling fluid filtrate tracer because it is can be recovered by Dean-Stark extraction of water from the core material. If ionic tracers are used instead of, or in addition to tritiated water, a selection criterion is that they should not be chemically or biologically altered by the reservoir rock. Anionic tracers used with varying degrees of success in water-based drilling fluid include bromide, iodide, and nitrate salts. Many reservoirs waters contain bromide and iodide in significant amounts, so accurate analysis of the reservoir water composition is important in selecting the appropriate tracer. The nitrate ion tracer requires a drilling fluid bactericide to prevent microbiological reduction from nitrate to nitrite.

Ionic tracers must be recovered from the cores by a method of water extraction, that requires considerably more time and

effort than extraction of tritiated water. The procedures are provided in 7.7. Ionic tracers provide semi-quantitative water invasion results.

d. Since the plug and donut samples are placed into the Dean-Stark distillation extraction apparatus in a frozen state, gas is prevented from escaping from the samples. Weight loss from gas release during extraction is included in the weight loss attributed to oil extraction. Oil is determined gravimetrically. If sufficient gas is present, a significant error may be introduced into the oil saturation determination. Thus, only water saturation is reported from filtrate invasion studies.

4.3.7.6 Accuracy/Precision

The precision of the measurements of the extent of drilling fluid filtrate invasion into the core is within about 5 to 20 percent. The simultaneous use of more than one drilling fluid tracer adds confidence in the reported filtrate invasion. There is no information on the accuracy of the method, since there is no accepted reference value for the extent of drilling fluid filtrate invasion downhole.

4.3.7.7 Calibration

Calibration of the liquid scintillation equipment, anionic tracer detection equipment, and ion chromatograph is essential for consistent results.

4.4 SOLVENT FLUSHING

4.4.1 Karl Fischer Titration

4.4.1.1 Principles of Analysis

Core plug samples are cleaned by dynamic miscible displacement with an appropriate sequence of solvents and the water content of the produced effluents analyzed by Karl Fischer titration (refer to ASTM D1364-90[6] and ASTM D4377-88[7]).

4.4.1.2 Apparatus

Apparatus appropriate for this method includes:

a. General—The apparatus includes a core flow system for the solvent injections and a Karl Fischer titrimeter for the effluent analysis. The core flow system is composed of a core holder assembly, solvent delivery device, and related hardware. The Karl Fischer analysis requires a titrimeter, appropriate Karl Fischer reagents, analytical balance, and syringes.

b. Core holder assembly—Hassler or hydrostatic core holder, sleeves, end-pieces, and a source of pressure appropriate for the expected confining and flow pressures.

c. Solvent delivery device—A constant rate high pressure liquid chromatographic (HPLC) pump or a gas-driven transfer system may be utilized. The pump may be a syringe or a continuous-flow type system. The constant pressure method requires a high pressure gas bottle, regulator, and a vessel such as a floating piston accumulator.

d. Related hardware—Consists of corrosion-resistant tubing, valves, and fittings to connect the core holder and solvent delivery system.

e. Karl Fischer titrimeter—Volumetric or coulomatic titrimeter.

f. Analytical balances—Covering range from 0.1 milligram to 1000 g.

g. Syringes—0.1–10 ml capacity, plastic or glass.

h. Materials and reagents:

1. Karl Fischer reagents: Utilize reagents appropriate for choice of titrimeter.
2. Toluene, reagent grade.
3. Methanol, Karl Fischer grade.
4. Glass vials, 25 ml capacity with caps lined with polytetrafluoroethylene (PTFE).
5. Glassware, stoppered vessels of approximately 1000 ml capacity, 10 ml receiving tubes.
6. Silver nitrate, 0.1 Normal.
7. Check solution, 0.1 weight percent water in methanol standard.

4.4.1.3 Procedures/Precautions

4.4.1.3.1 Procedures

This method is suitable for determining water saturation in whole core and plug-sized samples and can be used as an alternative to the distillation extraction (Dean-Stark) method.

a. The sample is weighed to the nearest 0.1 gram (assuming a plug-size sample), and loaded into the core holder. The desired confining stress is applied and any produced fluids are collected in a receiving tube and the volumes are recorded.

b. An alternating methanol-toluene solvent injection sequence is performed, beginning with the solvent that is miscible with the mobile phase, i.e., methanol if the brine is mobile or toluene if the oil is mobile.

c. All storage vials, syringes, or collection vessels should be weighed or tared prior to use.

d. A glass vial is filled with the initial injectant reagent and sealed. The solvent is then injected into the core sample at rates or pressures suitable for the core material, and the effluent is collected in a stoppered vessel. Care should be taken to minimize exposure of the solvent to atmospheric humidity.

1. For methanol injection, the effluent is sampled periodically and tested for the presence of salts using silver nitrate. Injection is complete when a drop of silver nitrate fails to form precipitates in the effluent aliquot.
2. For toluene, injection is continued until (a) the effluent appears clear, or (b) refractive index of the effluent equals the injectant.

3. The Karl Fischer titration is performed as soon as possible to minimize changes in water content due to absorption of moisture. (refer to Steps g–i).

e. Step d is repeated using the alternating solvent.

f. Step d is repeated using the initial solvent.

g. The storage vials and effluent vessels are reweighed and the weights of the solvents calculated.

h. Using a syringe, a portion of the injectant solution is analyzed for water content by Karl Fischer titration according to the manufacturers' recommended procedures. The syringe is reweighed to ascertain the exact weight of solvent analyzed. Making sure the effluent sample is uniform, water content is determined as described above. Results are reported in weight percent or ppm equivalent.

i. The sample is unloaded, dried using appropriate techniques, weighed, and the pore volume determined by helium injection.

4.4.1.3.2 Precautions

Precautions for Karl Fischer titration include:

a. Material safety data sheets should be consulted for proper handling procedures for all required chemicals, including the Karl Fischer reagents.

b. Methanol is highly susceptible to water absorption from atmospheric humidity as well as from untreated glassware. Since it is extremely difficult to quantify these variables, exposure to air should be minimized, and the Karl Fischer titrations performed as quickly as possible.

c. The Karl Fischer titration requires only a small amount of solvent; therefore, the analyzed sample must be representative of the entire solution to yield accurate results.

4.4.1.4 Calculations

The following calculations should be performed:

a. Water Saturation:

$$S_{wes} = W_{es} \times (S_{we} - S_{wi})$$

$$S_{ws} = \text{Sum } S_{wes}$$

$$S_{wb1} = S_{ws} / [(1 - A) \times \rho_b]$$

$$S_{wb2} = (S_{wb1} \times 100)/PV$$

Where:

ρ_b = density of brine, g/ml.
S_{wes} = water content of effluent solvent, g.
W_{es} = weight of effluent solvent, g.
S_{we} = effluent water content, g/g.
S_{wi} = injectant water content, g/g.
S_{ws} = sample water content, g.
Sum S_{wes} = summation of the water contents of the effluent solvents, g.
S_{wb1} = sample brine content, ml.
S_{wb2} = sample brine saturation, percent of the pore volume.
A = salt content of the brine, grams of salt per gram of brine.
PV = pore volume of the sample, ml.

b. Oil Saturation:

If the sample was fully saturated, oil saturation may be determined by volumetric difference:

$$S_o = 100 - S_{wb2}$$

If the sample contained an initial gas saturation, the oil saturation must be determined by gravimetric difference:

$$W_o = W_i - W_d - [S_{ws} / (1 - A)]$$

$$V_o = W_o / \rho_o$$

$$S_o = \frac{V_o \times 100}{PV}$$

Where:

S_o = oil saturation as a percent of the pore volume (PV).
S_{wb2} = sample brine saturation, percent of the pore volume.
W_o = weight of oil, g.
W_i = initial weight of the sample, g.
W_d = weight of the dry sample, g.
S_{ws} = sample water content, g.
V_o = volume of oil, ml.
ρ_o = density of oil, g/ml.
A = salt content of the brine, grams of salt per gram of brine.
PV = pore volume of the sample, ml.

c. Data reporting—Fluid saturations are reported to the nearest 0.1 percent pore space.

4.4.1.5 Advantages

Advantages of this method include:

a. All saturation levels can be determined.
b. The Karl Fischer titration is very precise.
c. Damage to sensitive minerals is minimized.
d. The procedure removes salts from the sample.
e. Saturations can be determined in special core tests in which stress hysteresis effects prohibit unloading the sample.

4.4.1.6 Limitations

Limitations of this method include:

a. Methanol readily absorbs moisture from the environment.
b. Accuracy of the method is dependent on solvent handling and storage techniques.
c. The analyzed sample must be representative of the bulk effluent.

d. The technique is more complex and expensive than the distillation extraction method.
e. Oil saturations are determined indirectly. Oil saturation by gravimetric difference assumes no grain loss of the sample.
f. This technique is not appropriate for samples that contain halite, sulfur, or other minerals that are soluble in methanol.

4.4.1.7 Accuracy/Precision

Precision of automated Karl Fischer titration units is ±0.5 percent of the measured value. However, the accuracy of the technique is limited to the ability to handle and store the solvents without altering the water content.

4.4.1.8 Calibration

Instrument operation is checked by analyzing standard solutions of known water content. Spiked samples should yield percent recoveries between 90 and 110 percent. Duplicate analyses are performed on 10 percent of the total samples analyzed and should have a relative percent difference less than 2 percent.

4.5 SCANNING METHODS

4.5.1 Introduction

Several laboratory techniques for scanning measurements of water, oil, and gas saturations in core samples have been reported. These techniques include: (a) linear x-ray absorption, (b) microwave absorption, (c) computer assisted (axial) tomography (CT), (d) linear gamma-ray absorption, and (e) nuclear magnetic resonance (NMR). The linear x-ray, gamma-ray, microwave, and CT methods may be considered as emerging technologies for saturation measurement. Other scanning techniques that have been used for saturation determinations are radio wave resonance and neutron attenuation radiography. The neutron radiography has a good potential for determination of fluid saturation because neutrons are attenuated much more by fluids (proton) than by rocks. Thus, neutron tomography may not require any addition of tagging agents to the resident fluids for saturation determination. These techniques are not widely used for core analysis.

4.5.2 Principles

The x-ray, CT, and gamma-ray techniques measure the absorption of high energy electromagnetic radiation by fluids that have been tagged with high absorption agents (usually high atomic weight elements) to determine fluid saturations. The microwave absorption technique is based on the absorption of microwave energy by water molecules. NMR is based on the detection of the proton (H1), carbon (C13), sodium (Na23), phosphorous (P31), and fluorine (F19) containing fluids by magnetic fields alternating at radio frequencies while the sample is located in a large constant magnetic field.

4.5.3 Apparatus

Apparatus for these techniques are generally sophisticated and expensive. X-ray and CT equipment manufactured for medical use are often adequate for saturation measurements. Medical NMR equipment is not often suitable for saturation measurements. Microwave and gamma-ray absorption apparatus have usually been specially built.

4.5.4 Procedures/Precautions

Several parameters must be considered for accurate saturation determination when scanning methods are used, such as the power level setting, tagging material and its concentration, tagging fluid-rock and fluid-fluid interaction (refer to the Bibliography for the many procedures and precautions for the individual techniques).

4.5.5 Calculations

Because all techniques (except NMR) generate signals from both the solid matrix and the fluids, measurements at a minimum of two known saturations (normally 0 and 100 percent) are required to establish a calibration line. The unknown saturations for the oil-brine system are then calculated based on the following equation:

$$S_w = (U_{sat} - U_o)/(U_w - U_o)$$

Where:

S_w = unknown brine saturation, as a fraction.
U_{sat} = attenuation of core at the unknown saturation (S_w).
U_w = attenuation of core at 100% brine saturation.
U_o = attenuation of core at 100% oil saturation.
(The units of U_{sat}, U_w, and U_o must all be the same.)

4.5.6 Advantages

An important advantage of these methods is their ability to provide information on the spatial distribution of the fluid saturation. Another advantage of these techniques is that the measurements are made noninvasively and nondestructively. Extraction of fluids is not required prior to flow tests on preserved cores (that can be difficult in full-sized or tight cores). Saturations can be monitored without terminating a laboratory experiment.

An advantage of CT and NMR imaging techniques is that they provide the saturation distribution of fluids in all three dimensions.

An advantage of the microwave and NMR techniques is their capability to determine water saturation in the core without addition of any tagging agents. The microwave and NMR techniques can be used for saturation determination in standard core analyses because they do not require any tagging agents and calibration curves are established after core cleaning and resaturation processes.

4.5.7 Limitations

A limitation of the x-ray, CT, and gamma-ray techniques is that they can determine saturations in cores only if they are saturated with tagged fluids. Therefore, they may not be suitable for the basic core analyses.

A limitation of the NMR technique is the inability to handle cores containing significant ferromagnetic materials, clays, or gas.

4.5.8 Accuracy/Precision

The accuracy and capability of some of the scanning techniques for saturation measurement are listed in Table 4-2. The accuracies listed in this table represent values achievable only under optimum calibration and measurement methodology.

4.5.9 Calibration

The absorption techniques require measurements of signal intensity at a minimum of two saturation levels to establish a calibration curve to relate instrument response to saturations. Calibration is instrument dependent.

4.6 COAL ANALYSIS

4.6.1 Principles of Analysis

Basic core analysis techniques, such as retort analysis and solvent distillation extraction, are not suitable for the determination of fluid saturations of coal samples. Alternative methods have been developed to measure the fluid saturation of coal. With coal, the fluid of primary concern is water. Water saturation, or more appropriately, moisture content is a fundamental property of coal that must be accurately determined to properly evaluate a coal seam. The method described here for determining the moisture content of coal involves drying water from a crushed sample in a convection oven and taking successive weight measurements until equilibrium is achieved. The weight loss is equivalent to the amount of water driven off, and moisture content is reported as the weight percentage of moisture with respect to the weight of the wet coal sample.

4.6.2 Apparatus

The following apparatus are appropriate for this method:

a. Crusher—Use a jaw, cone, or rotary crusher to reduce the coal to the size retained on a No. 8 (2.38 millimeter) sieve.

b. Drying pans—The pans used should be of sufficient size to accommodate crushed samples spread to a thickness of less than 2.5 centimeters. The sides of the pans should not be greater than 3.8 centimeters in height. The pans should be made of a material that is stable and will not corrode at the temperature used.

c. Drying oven—A forced-draft oven that is capable of maintaining a constant temperature of 225°F, ±5°F (107°C, ±3°C) should be used. The velocity of forced air in the oven should be sufficiently low so as not to disturb the coal sample. An oven with a gas port capable of accommodating a hose from a nitrogen tank is desirable, because flowing nitrogen, rather than air, through the oven can help minimize oxidation of the coal sample.

d. Balance—The balance used to weigh the samples should be sensitive to 0.1 gram and have a capacity large enough to accommodate both the sample and the pan.

e. No. 8 Sieve (2.33 millimeter)—The sieve should be large enough to hold the entire prepared sample while sifting out the larger particles.

f. Glass desiccator—The desiccator should be large enough to hold the prepared sample and sample pan. It is recommended that either no desiccant or fresh color-coded desiccant be used because some desiccants actually give off moisture if not fresh.

4.6.3 Procedures/Precautions

4.6.3.1 Sampling

4.6.3.1.1 Crushing

Use a jaw crusher or other suitable type of crusher or instrument to reduce the coal to the size retained on a No. 8 (2.38 millimeter) sieve. At least 95 percent of the crushed sample should pass through a No. 8 sieve.

Table 4-2—Measurement Capabilities of Scanning Saturation Techniques

Technique	Dimensions	Tagging requirements	Accuracy, saturation units	Spatial resolution, cm	Maximum sample size, in.	Measured fluids
Linear x-ray	2	yes	1	1.0	12	oil, water, gas
Computer Assisted Tomography	3	yes	1	0.2	12	oil, water, gas
Gamma-ray	2	yes	3	0.1	12	oil, water, gas
Microwave	2	no	1	2.0	2	water
Nuclear Magnetic Resonance	3	no	1	0.1	4	oil, water

4.6.3.1.2 Sample Size

The sample used for moisture content determination should have a minimum weight of about 500 grams.

4.6.3.2 Weighing

4.6.3.2.1 Drying Pan

Immediately after sample preparation is complete measure and record the weight of the clean and dry drying pan. Pour the crushed, sifted coal into the drying pan and spread to a thickness of not more than 2.5 cm.

4.6.3.2.2 Sample Initial Weight

Measure and record the weight of the wet coal sample and the drying pan. Place the pan in the oven at a temperature of 225°F, ±5°F (107°C, ±3°C).

4.6.3.2.3 Dry Weight Measurements

After the sample has dried for a period of 1 hour, remove it from the oven and place it in a glass desiccator to cool. The use of desiccant is not recommended. Weigh the sample immediately after cooling and record the weight. Return the sample to the oven. Repeat this weighing procedure at half-hour intervals until the weight loss between successive measurements is less than 0.05 percent of the sample weight. The final measurement taken is considered the dry weight of the sample plus the weight of the drying pan.

4.6.4 Precaution

Do not over-dry the coal sample. Oxidation of the coal occurs if it dries too long in air.

4.6.5 Calculations

Calculate moisture content using the following equation:

$$M = [(W_2 - W_3)/(W_2 - W_1)] \times 100$$

Where:

M = moisture content of coal sample,%.
W_1 = weight of empty pan, g.
W_2 = weight of wet coal plus pan, g.
W_3 = weight of dry coal plus pan, g.

4.6.6 Advantages

The principle advantage of this method is its simplicity. The procedure does not require a large capital investment in specialized equipment. Neither does it require specialized training to perform the measurements. More sophisticated techniques are available to determine the moisture content of coal (refer to 4.10, References 8, 9, and 10). However, in the context of basic core analysis, the technique described here is adequate.

4.6.7 Limitations

Some types of coals have a tendency to oxidize using the technique described here. Moisture content results will be too low if oxidation occurs; oxidation adds weight and will cause the dry weight to be too high. It is recommended that drying times be kept within the guidelines outlined here to minimize the possibility of oxidation. Forcing nitrogen, rather than air, through the oven may also help to reduce the potential for oxidation.

4.6.8 Accuracy

The accuracy of this technique is very good because it depends only upon the accuracy of the balance. Thus, the accuracy for this technique is ± 0.05 percent.

4.6.9 Precision

Results of duplicate moisture content determinations performed on the same sample by the same technician using the same equipment should not differ by more than ±0.3 percent. Results of moisture content measurements performed by different laboratories on adjacent samples should not differ by more than ±0.5 percent.

4.6.10 Calibration

Use of a calibration weight set is recommended to ensure that the balance works properly. Periodic calibration of the balance at regular intervals should also be performed by a trained technician according to factory specifications to ensure measurement accuracy.

4.7 OIL SHALES

4.7.1 Principles of Analysis

Oil shale is defined as rock composed of clay to silt size particles containing various quantities of solid organic material (e.g., kerogen). The solid organics present in these samples are typically liquefied to generate oil. Analysis of this type of formation, with low permeability and potential for solid organics, requires special procedures.

Oil and water saturations are obtained by a high temperature retorting process. No attempt is made to measure gas-filled pore volume. The fluid saturations are reported in gallons per ton.

4.7.2 Apparatus

The apparatus is the same as used for the Retort Method at Atmospheric Pressure (see 4.2.1.2). The fluids are collected in 15 cm^3 centrifuge tubes. Typically, gas is evolved during the retorting process. Should it be desirable to collect/measure these gases, modified glassware may be used consisting of a calibrated liquid receiving section with the upper portion

of the glassware fitted with a sidearm to allow produced gases to be channeled to collection cells or measuring equipment.

4.7.3 Procedures/Precautions

The procedure is similar to the procedures described in 4.2.1.3, with the following exceptions:

a. The representative sample, approximately two inches in length, is not broken in half. Permeability is a not a consideration in oil shale analysis.

b. No attempt is made to screen the crushed sample to exclude fines created during the crushing process.

c. An adjacent sample is not prepared for the mercury pump tests described in 4.2.1.3, because neither the gas-filled pore volume nor the porosity are required.

d. Oil coking and cracking losses are ignored. If significant, a correction for mechanical oil hold-up should be made.

e. The oven is allowed to heat at a rate consistent with the heating elements and number of retorts in place, i.e., no attempt is made to measure pore water at an initial oven temperature setting. The oven temperature is set initially at 1000°F (538°C). When the oven has stabilized at this temperature and all collection tube volumes remain constant, the retorting process is considered complete.

f. Under certain conditions (e.g., samples containing different varieties of kerogen) the volume of oil recovered may vary with the rate of heating.

g. Only the total water collected is used for the final calculation of water content.

h. Retorting operations should be carried out with adequate ventilation (e.g., under fume hoods), as typical oils recovered from kerogens are high in sulfur content and give off noxious vapors.

4.7.4 Calculations

The oil and water contents are calculated as follows:

$$\text{Oil Content} = \frac{\text{Volume of Oil Recovered} \times 239.7}{\text{Sample Weight}}$$

$$\text{Water Content} = \frac{\text{Volume of Water Recovered} \times 239.7}{\text{Sample Weight}}$$

Where:

Oil volume is in milliliters.

Water volume is in milliliters.

Sample weight is in grams.

Oil content is in gallons/ton.

Water content is in gallons/ton.

4.7.5 Advantages

Advantages of this method include:

a. Large volumes of sample can be used.
b. The method is fairly rapid.
c. All measurements are made directly and independently of others.
d. Standard retorting equipment and procedures can be used.
e. Care is not required to differentiate pore water from total water.
f. Oil volume correction curves are not required.
g. Duplicate samples are more easily obtained since a large volume of crushed material may be prepared, and samples obtained, by the “cone and quarter” technique.

4.7.6 Limitations

Limitations of this method include:

a. Oil yields may be dependent on rate of heating.
b. Due to emulsions, there may be difficulty in reading the meniscus in the collection glassware.

4.7.7 Accuracy/Precision

Accuracy/precision of this method is:

a. The accuracy of the oil and water volumes is ±2.5 percent of the measured volumes.
b. The reproducibility should fall in the ±0.5 gal/ton range for low yield samples (e.g., 3 to 10 gal/ton) and ±1.0 gal/ton in high yield samples (e.g., 20 to 40 gal/ton).

4.7.8 Calibration

For glassware calibration procedures, refer to 4.2.1.8.

4.8 GYPSUM-BEARING CORES

4.8.1 Principles of Analysis

Cores containing gypsum ($CaSO_4.2H_2O$) often are from carbonate formations. Such formations are frequently heterogeneous with vugs and/or fractures so that full-diameter core analysis procedures are preferred. However, determination of fluid saturations of gypsum-bearing cores by the distillation extraction method (Dean-Stark) for full diameter cores (see 4.3.2) using toluene as the solvent is not recommended. At the boiling point of toluene, gypsum dehydrates. If the core contains a significant amount of gypsum, erroneously large water volumes and incorrect pore volumes will be measured. The pore volume used to determine saturation will also be in error because of the increase in pore volume due to the dehydration of the gypsum. The volume of oil extracted from the sample is not affected by the presence of gypsum.

There are emerging technologies, e.g., NMR that show promise for the quantification of gypsum and in the future

may be offered as basic services by commercial laboratories. The Karl Fischer titration method (see 4.4.1) can be used to analyze gypsum-bearing cores by determining water content of flushed solvents during cool solvent extraction. However, this method is generally not available at service companies for analysis of a large number of core samples. The qualitative method described in the following paragraph can be performed by commercial laboratories.

The qualitative method presented here is based on adjustment of retort saturations for the dehydration of the gypsum by comparing retort and low temperature porosities and weights on adjacent samples. A plug sample is cleaned by pressure cycling solvents at low temperature so that oil and water are extracted from the sample without removing the chemically-bound water held within the gypsum. Boyle's law porosity is then measured on the cleaned sample. After the Boyle's law porosity is determined, the plug sample is retorted to determine the amount of bound water in the sample. The difference in porosity between the low and high temperature extractions is used to calculate the bulk volume of gypsum contained in the core sample. The volume of water given off in the dehydration of gypsum is approximately 1.27 times the increase in pore volume that results from shrinkage of gypsum (grain density = 2.32 g/cm^3) as it converts to anhydrite (2.89 to 2.98 g/cm^3). Retort saturation measurements are obtained on trimmed ends of the plug sample or on material adjacent to where the plug was drilled. The retort method at atmospheric pressure (see 4.2) removes the fluids in the core, and water of crystallization, from the gypsum. The summation-of-fluids porosity is determined from the retort data. Water saturations determined by the retort method are then adjusted. The adjustment will result in a decrease in water saturation. This adjustment is qualitative because carbonates are noted for being heterogeneous; so the companion samples may not contain the same amount of gypsum.

4.8.2 Apparatus

Appropriate apparatus for this method include:

a. High temperature retorting equipment (see 4.2.1.2).

b. Low temperature cleaning apparatus, e.g., pressure saturator large enough to pressure cycle a solvent through several samples at one time.

c. Boyle's law porosimeter.

4.8.3 Procedures

The core to be analyzed should be laid out on a table and fitted together as in standard practice (see 3.2.4). From each foot of core to be analyzed, drill a plug sample. If 1-inch diameter plugs are used, the samples should be at least one inch long. After the plugs are trimmed to length, they should be weighed. Check fluorescence of the plug and record. The initial visual estimate of hydrocarbon saturation will allow the analyst to better evaluate the sample during the pressure-cleaning phase. The plug should be cleaned by a low temperature method that minimizes gypsum alteration.

The fluid saturation of the trimmed plug ends and/or the remaining material from the segment of core from which the above mentioned plug has been drilled, is determined by the retort method at atmospheric pressure (see 4.2.1.3). Use the maximum amount of core material possible for the best precision. This will yield high temperature fluid saturation data and the summation-of-fluids porosity may be calculated (see 5.3.2.2.2 and 4.2.1).

The drilled plug samples should be cleaned in a pressure vessel using an azeotrope mixture of $^1/_3$ acetone, $^1/_3$ cyclohexane, and $^1/_3$ methanol. The samples are placed in the pressure vessel and totally immersed in the azeotropic mixture. Air or nitrogen pressure is applied to the top of the mixture forcing the azeotrope into the pore spaces of the plug samples. The pressure is cycled to force the mixture in and out of the plugs. After approximately 24 hours, the fluorescence of the samples should be checked to see if extraction is complete. Plugs with oil fluorescence should be returned to the pressure vessel for another cleaning cycle. If there is no visible oil fluorescence, the samples are deemed clean. The samples are placed in a vacuum oven at 120°F (49°C) until weight stabilizes. Samples should stay in the vacuum oven for at least 24 hours to remove the azeotrope mixture from the samples. Low temperatures are used during the cleaning and drying phases of this process to minimize alteration of gypsum in the plug samples. After the plug samples have been cleaned and dried, Boyle's law porosity is determined.

After the Boyle's law porosity is measured on the plug sample, the plug is retorted to remove the water bound in the gypsum. After retorting, the plug is reweighed and the porosity of the plug sample is remeasured by Boyle's law. Comparison of the retort porosity with the low temperature porosity identifies zones with gypsum. When the high temperature porosity is much greater than the low temperature porosity, the gypsum content is very high. When the two porosities agree, the gypsum content is low.

The oil volume extracted by the retort method is not affected by the presence of gypsum. However, the pore volume used to calculate the oil saturation must be corrected for the presence of gypsum. The oil saturation can be calculated from the corrected pore volume and the oil volume.

4.8.4 Precautions

Care should be exercised drying samples taken from the pressure saturator to prevent dehydration of any gypsum present. Monitor vacuum oven heat with a temperature probe.

4.8.5 Calculations

Refer to Retort Method at Atmospheric Pressure (see 4.2) and Summation-of-Fluids Porosity Calculations (see

5.3.2.2.2). Refer to 5.3.2.1.1, "Boyle's Law Porosity Calculations".

$$S_{oc} = V_o / (V_{br} \times \phi_{lt})$$

Where:

V_o = volume of oil obtained from the retorting of the trimmed ends or material adjacent to the plug sample, cm^3.

V_{br} = bulk volume of the retorted material, cm^3.

ϕ_{lt} = porosity of the plug after low temperature extraction, fraction.

S_{oc} = adjusted oil saturation, fraction.

If the gas saturation is assumed to be zero, then the water saturation, S_{wc}, is:

$$S_{wc} = 1 - S_{oc}$$

If the gas saturation is non-zero, the water saturation can be calculated by assuming a grain density for anhydrite:

$$\Delta V_w = (\rho_a / \rho_g) \times (V_{br} \times \Delta\phi)$$

$$\Delta_{wcorr} = V_w - \Delta V_w$$

$$S_{wc} = V_{wcorr} / (V_{br} \times \phi_{lt})$$

Where:

Swc = water saturation adjusted for the gypsum dehydration, fraction.

ΔV_w = volume of water from the dehydration of the gypsum, cm^3.

ρ_a = grain density of anhydrite, g/cm^3.

ρ_g = grain density of gypsum, g/cm^3.

$\Delta\phi$ = difference in porosity between the high and low temperature extractions of the plug sample, fraction.

V_{wcorr} = volume of water adjusted for the gypsum dehydration, cm^3.

V_w = Volume of water obtained from the retorting of the trimmed ends or material adjacent to the plug sample, cm^3.

Alternatively, the saturations can be expressed in terms of sample weights:

$$f_{wat} = W_{wat} / (W_{wat} + W_{oil})$$

$$W_{ewat} = (W_{po} - W_{pr}) \times f_{wat}$$

$$W_{eoil} = (W_{po} - W_{pr}) \times (1 - f_{wat})$$

$$W_{gyp} = W_{pl} - W_{pr}$$

$$V_{wcorr} = (W_{ewat} / \rho_{br}) - (W_{gyp} / \rho_{gw})$$

$$S_{oc} = (W_{eoil} / \rho_{oil}) / V_{plt}$$

$$S_{wc} = V_{wcorr} / V_{plt}$$

Where:

W_{po} = weight of the plug after drilling, g.

W_{pl} = weight of the plug following low temperature extraction, g.

W_{pr} = weight of the plug following retorting, g.

W_{wat} = weight of water determined by the retort extraction of the end pieces or adjacent material, g.

W_{oil} = weight of the oil determined by the retort extraction of the end pieces or adjacent material, g.

f_{wat} = weight fraction of water removed by retort extraction, fraction.

W_{ewat} = estimated water content of the plug sample including the bound gypsum water, g.

W_{eoil} = estimated oil content of the plug sample, g.

W_{gyp} = gypsum water of hydration in the plug sample, g.

ρ_{br} = density of the formation brine, g/cm^3.

ρ_{gw} = density of the gypsum bound water, g/cm^3.

V_{wcorr} = volume of water adjusted for the gypsum dehydration, cm^3.

ρ_{oil} = density of the formation oil, g/cm^3.

V_{plt} = pore volume of the plug sample following low temperature cleaning, cm^3.

S_{oc} = oil saturation calculated for the plug sample accounting for the change in pore volume due to the dehydration of the gypsum, fraction.

S_{wc} = water saturation calculated for the plug accounting for the bound gypsum water and the change in pore volume due to the dehydration of the gypsum, fraction.

4.8.6 Advantages

Advantages of this method include:

a. Damage to gypsum is minimized when the sample is cleaned by pressure cycling. Pore volume measurements used to calculate saturations are more representative if the gypsum is not damaged.

b. Gypsum content can be estimated by porosity comparisons.

4.8.7 Limitations

Limitations of this method include:

a. The low temperature cleaning process can be time consuming depending on the oil gravity and the permeability of the core material.

b. The retort oil and water saturation values are not as precisely determined as for samples that contain no gypsum.

4.8.8 Accuracy/Precision

Accuracy/precision of this method is:

a. If the water of crystallization is preserved during pressure cycling, pore volume determination will have the standard accuracy.
b. The precision of oil and water saturation calculations determined by retort method will be reduced due to the estimation of gypsum content by porosity comparisons.
c. Porosities obtained by the Boyle's Law helium method are more reliable than those obtained by the summation-of-fluids method. When porosity values obtained by Boyle's Law and summation-of-fluids methods are in good agreement, the saturations are assumed to be more reliable than in intervals in which there are large differences between the two porosities.

4.8.9 Calibration

The equipment used to measure porosity by both methods must conform with standards for the appropriate porosity method.

4.9 HISTORICAL

4.9.1 Vacuum Retort Method

The vacuum retort method for obtaining core fluid saturations is a full-diameter technique. This method is used extensively in "hard rock" regions, e.g., West Texas, where production is predominately from carbonate formations. Although several core analysis companies still maintain equipment for this procedure it is not a recommended procedure, having been replaced by the distillation extraction method (see 4.3).

The procedure consists of distilling off the pore fluids at a maximum temperature of 450°F (232°C) while maintaining a partial vacuum on the system. The fluid collection glassware should be immersed in an alcohol/dry ice bath maintained at approximately –75°F (–59°C) to prevent the vapors from being lost through the vacuum pump.

The procedure lends itself to the determination of the pore volume by the summation-of-fluids method. This is possible because the first step consists of saturating the unoccupied or gas-filled pore spaces with fresh water. The unoccupied pore volume is determined by gravimetric difference. An oil volume correction factor is employed and the pore water is determined by difference. The three values comprise the pore volume and the collected fluids can be expressed accordingly. A porosity can be calculated with a bulk volume determined on the same sample used for the distillation process.

One of the prime disadvantages of this method is the condition of the cores after the distillation process. It is not uncommon for the cores to be black from coked oil and no cleaning process will restore the samples to a condition whereby they could be deemed suitable for use in further testing procedures, e.g., a Boyle's Law porosity, capillary pressure test, relative permeability, etc. For this and other reasons, the authoring Subcommittee decided not to record this as a recommended procedure.

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5 Porosity Determination

5.1 GENERAL

5.1.1 Introductory Comments/Equations

5.1.1.1 Porosity, which is defined as a ratio of the void-space volume to the bulk volume of the material, is an intrinsic property of all reservoir rocks. The amount of void space which can be occupied by hydrocarbons or water in a reservoir must be known for an intelligent estimate of the initial oil/gas in place. The precision with which porosity can be determined is largely a function of the methods used in these measurements. Several logging tools using either electrical, nuclear, density, or sonic methods are used for continuous porosity determination in the wellbore. The measurements obtained from these well logging tools should be calibrated against core porosities determined under simulated subsurface conditions, bearing in mind the volume of rock investigated by logging tools. Effects of core sample bias, sample frequency and sample volume must be taken into consideration. With one exception, all discussions refer to porosity determination at zero to <400 psi (2760 kPa) confining stress. The method to determine porosity at simulated overburden pressure is discussed in 5.3.2.2.1.2.

5.1.1.2 Unless otherwise stated, the analysis methods described are applicable to cylindrical core plugs drilled from consolidated and relatively homogeneous rock. Porosity from core analysis may commonly be arrived at by several different methods (see Table 5-1). Measurement of bulk volume (*BV*) and grain volume (*GV*) yields pore volume (*PV*) by difference, and porosity (ϕ) by:

$$PV = (BV - GV) \tag{1}$$

$$\phi = (BV - GV) / BV$$

Direct measurement of pore volume (*PV*) and grain volume (*GV*) yields:

$$\phi = PV / (PV + GV) \tag{2}$$

Direct measurement of pore volume (*PV*) and bulk volume (*BV*) yields:

$$\phi = PV / BV \tag{3}$$

5.1.1.3 In the laboratory, usually one of two types of porosity are measured; viz., effective porosity or total porosity. There is a difference between the historical definitions of these porosities and the current definition used by log analysts. This is further complicated by core analysis techniques designed to leave several molecular layers of clay- or mineral-bound water within the pore space (see Figure 5-1).

5.1.1.4 Historically, effective porosity has been defined as a measure of the connected voids. It is derived from either the difference between bulk volume and apparent grain volume determinations, or by a direct measurement of the connected void space. The measured volume of the connected void space may vary with sample preparation and the analytical method used. Total porosity is a measure of the total void space, both connected and isolated, in the rock sample. This can be determined by bulk volume, dry weight, and grain volume measurement on a disaggregated sample.

5.1.1.5 In the field of log interpretation, effective porosity has been defined as interconnected pore volume occupied by free fluids. In this definition, effective porosity does not include the volume of clay-bound or mineral-bound water, and in some analytical approaches, does not include the additional water held within the shale by capillary forces. Also in the field of log interpretation, total porosity is that volume occupied by all fluid (connected and isolated pore space) in the rock, including the volume occupied by the clay-bound water (see Figure 5-1).

5.1.1.6 Core analysis experience indicates that for most reservoir rocks there are few isolated pores, and hence there is very little or no measurable difference in historically defined effective and total porosity (see Figure 5-1). Thus, with these definitions, effective porosity determined from core analyses on a totally dried sample at 210° to 240°F (99° to 116°C) more closely corresponds to the total porosity as defined by the log analysts. In certain circumstances, such as volcanic rocks, there can be a measurable difference between the effective (connected) and total (connected and isolated) porosity from core analysis. In addition, core humidity drying techniques can be tailored to leave some amount of bound water on mineral surfaces.

5.1.1.7 There is no universal agreement between log analysts on the definition of effective porosity (see Figure 5-1). The lowest value for this parameter is obtained if all shale-associated water (adsorbed/anion free water plus capillary-held water within the shale) is excluded as noneffective pore space. A higher value is obtained if only the adsorbed/anion-free water (defined as a function of both salinity and cation exchange capacity or the concentration of clay counterions per unit pore volume, meq/cm^3) is excluded. Core porosities based upon humidity-controlled, dried samples are normally between this higher value and the total porosity. At this time, it is difficult to select laboratory conditions that yield effective porosity on core samples as defined by log analysts.

5.1.1.8 Another facet of porosity measurement, which in fact applies to the entire field of core analysis, is the selection of conventional (small sample) analysis versus full-diameter

analysis. The decision as to which type should be used would ideally be based entirely on the homogeneity of the formation being analyzed. Many sandstones are sufficiently homogeneous so that a small sample can be considered representative of the analysis increment. On the other hand, when the formation is heterogeneous as to pore structure or lithology, such as in vugular or fractured carbonates, full-diameter core techniques are more appropriate. Therefore, the size of the sample required to adequately represent the pore structure and lithology of the core material, as well as the sampling strategy should control the type of analysis used.

5.1.2 Poorly Consolidated/Unconsolidated Core Samples

5.1.2.1 Poorly consolidated/unconsolidated samples present unique requirements. The term "poorly consolidated" encompasses a wide range of core materials, from friable samples to completely unconsolidated samples with no apparent cementation between grains. For the purposes of this discussion, "poorly consolidated" refers to any plug sample that requires mounting in an appropriate jacketing material to withstand the cleaning, preparation, and measurement process.

5.1.2.2 Plug samples are commonly jacketed using a cylinder of metal or polymer about the circumference of the sample. Table 5-2 lists some common jacketing material along with their advantages and disadvantages.

Table 5-1—Bulk, Grain, and Pore Volume Directory

Method Number	Title	Purpose
5.2.1	Archimedes (Buoyancy) Mercury Immersion	Bulk volume measurement
5.2.2	Mercury Displacement	Bulk volume measurement
5.2.3	Caliper	Bulk volume measurement
5.2.4	Archimedes (Buoyancy) with Fluids Other than Mercury	Bulk volume measurement
5.2.5	Bulk Volume by Summation of Grain and Pore Volume	Bulk volume determination
5.3.1	Total Pore Volume from Disaggregated Sample Grain Density	Grain volume measurement
5.3.2.1.1	Boyle's Law Double-Cell Method	Grain volume measurement
5.3.2.2.1	Boyle's Law Single-Cell Method	Direct pore volume measurement
5.3.2.2.2	Summation of Fluids Porosity	Summary bulk and pore volume measurements
5.3.2.2.3	Liquid Saturation Method	Direct pore volume measurement
5.4	Historical Procedures	Bulk, grain and pore volume measurements
5.5	Organic Rich Rocks	Pore volume and adsorbed gas

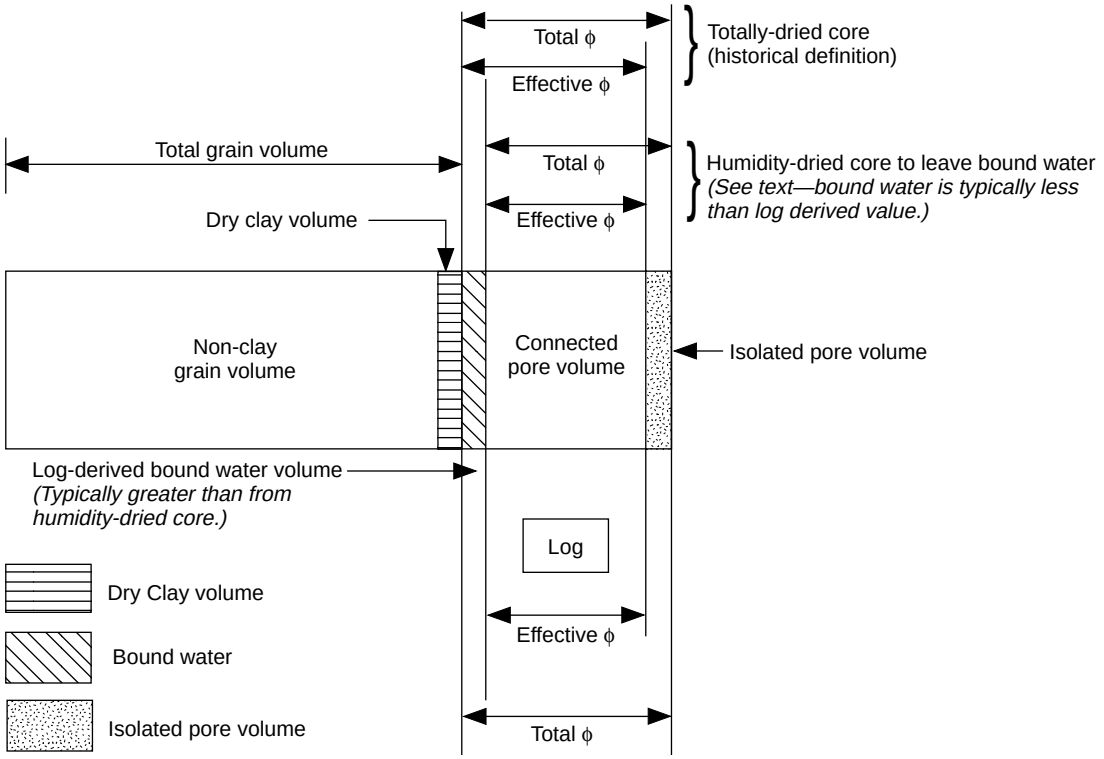


Figure 5-1—Total and Effective Pore Volumes as Defined by Core and Log Analysts

Table 5-2—Jacketing Material for Poorly Consolidated/ Unconsolidated Core Samples

Jacketing Material	Advantages	Disadvantages
Lead Sleeves	Malleable, conforms well to sample	Interacts with mercury and some brines
Teflon® tape	Inert	Difficult to apply, porous and permeable
Heat shrink Teflon tubing	Inert, easy to apply	May not conform well to sample at low confining stress, possible sample alteration from heat and radial stress applied as tubing shrinks
Aluminum sleeve	Malleable, conforms reasonably well to samples	Interacts with some brines and may not conform well at low confining stress
Paint-on epoxy or other coating	Cheap, easy to apply	Interacts with solvents, poor mechanical strength, can imbibe into sample
Tin	Malleable, conforms well to sample	Interacts with mercury and some brines

5.1.2.3 To allow flow, the plug faces are covered with screens or other porous material. The mesh size of the screens must be small enough to prevent grain loss, but large enough to avoid plugging by mobile fines. Screens with a mesh size of 200 or 120 are typically used. Samples with some consolidation require only a single screen. Less consolidated samples may require more sophisticated end treatments. For instance, two screens may be used. One screen is of finer mesh to prevent grain loss, and a second less flexible, coarser screen is added for mechanical strength. The material chosen for the screens must be inert to the solvents and brines used for cleaning and testing. A high quality stainless steel such as 316 or Monel is recommended.

5.1.2.4 The apparent pore and grain volume of the jacketing material and end screens must be determined and accounted for in the porosity measurement. The volume of jacketing material on an individual sample can be determined by applying the Boyle's Law Double-Cell Method for grain volume (see 5.3.2.1.1) or the Archimedes method for bulk volume using either mercury (where suitable, see 5.2.1) or other solvents (see 5.2.4). In practice, it is easier to determine the density of the jacketing material and then determine the weight of jacketing material applied to each sample. Jacket volume can then be computed. This method assumes that the density of the material is constant from piece to piece.

5.1.2.5 The "grain" and "pore" volume of the screens must be determined as well. The grain volume can be determined by taking a number of identical screens and measuring their solid volume in a Boyle's Law Double-Cell apparatus. The value determined is then divided by the number of screens tested and this average value is applied to each sample. The pore volume is more sensitive to the test method applied. For the Boyle's Law method, the pore or void volume in the screens can be determined by mounting a solid non-porous plug in the same manner as the real samples are prepared. The Boyle's Law Single-Cell method will then yield the screens' void volume.

5.1.2.6 Once a sample is packaged with a jacketing material and screens, it can be tested using many of the same techniques applied to consolidated samples. In the following sections, comments about the applicability of each measurement method for jacketed poorly consolidated samples will be made in 5.2.1.3.

5.1.2.7 Since the physical properties of poorly consolidated samples are more sensitive to confining stress than consolidated samples, measurements performed at stresses below in-situ effective reservoir stress will not be as representative of the reservoir rock. Therefore, the recommended method for determining the porosity of poorly consolidated samples is to use the Boyle's Law Single Cell method for direct pore volume measurement at elevated confining stress (see 5.3.2.2.1.2), and the Boyle's Law Double-Cell method for grain volume (see 5.3.2.1.1).

5.2 BULK VOLUME MEASUREMENT (PLUG SIZE)

The bulk volume of a rock sample is required to determine the porosity of that sample. Bulk volume of a core plug can be determined by several methods. Measurement techniques include Archimedes immersion, mercury displacement, and calipering.

Bulk volumes can also be calculated by summing direct measurements of grain volume and pore volume.

The bulk volume of a sample selected for porosity measurement should preferably be at least 10 cm^3. Normally, samples are a right cylinder with diameters from 2.54 cm to 3.81 cm and lengths of at least 2.54 cm and 3.81 cm, respectively. Irregular shapes may be used with proper precautions if samples of regular dimensions cannot be obtained.

5.2.1 Archimedes (Buoyancy) Mercury Immersion

5.2.1.1 Principle

A core plug is immersed in mercury and the volume of mercury displaced by the sample is determined gravimetrically (Archimedes' principle).

5.2.1.2 Advantages

Advantages of this method include:

a. Samples can be used for subsequent tests if no mercury penetration occurs.
b. The method is very accurate if careful technique is used and precise measurements are made.

5.2.1.3 Limitations

Limitations of this method include:

a. Trapping air around the samples will create errors and yield erroneously high bulk volumes.

b. Vuggy samples or extremely high permeability samples will be penetrated by the mercury resulting in low bulk volume values and rendering the samples unsuitable for further testing. Samples with a vugular surface or containing open fractures are not recommended for bulk volume analysis by mercury immersion. However, if it is necessary to analyze such samples using this technique, the sample surface must be sealed by coating or filling the vugs to prevent mercury penetration. This may not be an adequate treatment.

1. Materials such as heat-shrink Teflon tubing can be used for coating the cylindrical surfaces of a sample to cover the vugs. The volume of any coating must be subtracted from the total bulk volume measured, and air gaps on nonvugular surfaces must not exist between the tubing and the rock.
2. The surface vugs can be filled (e.g., with modeling clay, paraffin, or an epoxy) which will allow a direct measurement of the bulk volume when the sample is immersed under the mercury. Usually, filling the vugs is a destructive test and should be the last step in the analysis.
3. Calipering sample length and diameter, and computing bulk volume (see 5.2.3), is a method utilized for vuggy samples. Experience has shown that computed bulk volumes are often too high. Calibration techniques presented in 5.2.3.6 improve data accuracy.

c. The method is unsuitable for poorly consolidated/unconsolidated samples that are mounted in lead sleeves because of lead-mercury amalgamation. Mercury may also be trapped between jacketing materials and the sample; consequently, this is not a recommended procedure for jacketed samples.

5.2.1.4 Accuracy

The measurement of weight can be repeated within ±0.015 grams if air is not trapped when the sample is submerged and the temperature remains constant. Bulk volume measurements can be repeated to within ±0.01 cm^3.

5.2.1.5 Apparatus

The following apparatus are appropriate for this method:

a. Electronic, single-pan, analytical balance accurate to ±0.01 gm.

b. Mercury vessel large enough that plug positioned with the large dimension horizontal can be fully immersed without touching the sides of the container.

c. Mercury to fill the vessel so that the plugs can be fully immersed.

d. Adjustable, pronged fork with reference mark.

e. Thermometer.

5.2.1.6 Procedure

Bulk volume (BV) is measured by the Archimedes Mercury Immersion method with the apparatus shown in Figure 5-2. A cup of mercury is placed on a single-pan electronic balance, and a pronged fork is immersed to the fork reference mark. The mark is located such that the top of the core plug will be submersed 3 to 7 millimeters under the mercury. The balance is then tared. The fork is withdrawn from the mercury, and the plug to be measured is floated lengthwise on the mercury surface. The plug is then forced into the mercury with the fork to the same reference mark. The sample must not touch the side of the mercury vessel. The resulting weight represents the mass of the mercury displaced. Eighteen-gauge

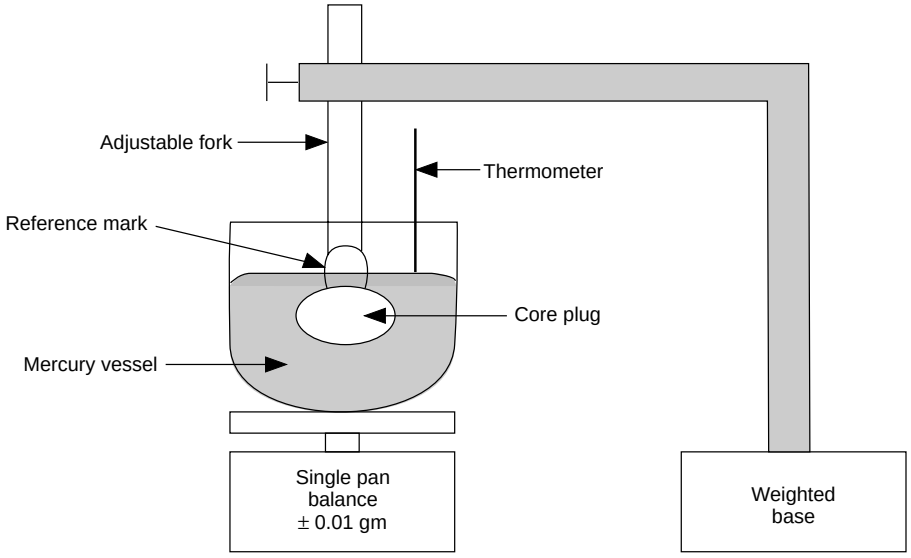


Figure 5-2—Archimedes Mercury Immersion Apparatus

(1.0-millimeter diameter) stainless steel wire is a suitable material for the fork. Heavier gauge wire has been observed to result in unstable weight measurements (changing weights with time) and long balance equilibrium times.

5.2.1.7 Calculations

The bulk volume is calculated using the following equation:

$$BV = \frac{\text{Mass of mercury displaced}}{\text{Density of mercury at measurement temperature}} \quad (5)$$

5.2.1.8 Precautions

A heavy weighted base for supporting the fork assembly is essential to hold the sample firmly in place to ensure accurate weights. It is important to use the correct mercury density for the mercury temperature. A temperature variation of 5°C will induce a systematic error of 0.02 percent in the bulk volume (see Table 5-3).

Table 5-3—Density of Mercury Versus Temperature[a]

°C	Hg Density gm/cm³	°C	Hg Density gm/cm³
18.0	13.5512	27.0	13.5291
19.0	13.5487	28.0	13.5266
20.0	13.5462	29.0	13.5242
21.0	13.5438	30.0	13.5217
22.0	13.5413	31.0	13.5193
23.0	13.5389	32.0	13.5168
24.0	13.5364	33.0	13.5144
25.0	13.5340	34.0	13.5119
26.0	13.5315		

[a]From *Handbook of Chemistry and Physics*, 71st Edition, 1990-1991, Editor-in-Chief: David R. Lide, Ph.D., CRC Press, Boca Raton, Florida.

In order to obtain stable weights, the fork should be of such design as to yield point contact on the sample. If the fork volume is too large relative to the plug volume, erroneous weights will be measured. Unstable weights may indicate an unsuitable fork or mercury invasion. Mercury contamination can be confirmed by comparing sample weights before and after mercury immersion.

5.2.2 Mercury Displacement (Volumetric Displacement Pump)

5.2.2.1 Principle

Sample bulk volume is measured by mercury displacement utilizing a volumetric displacement pump to which a stainless-steel sample chamber is attached.

5.2.2.2 Advantages

Advantages of this method include:

a. This procedure allows very rapid measurements.

b. The technique is employed as part of the Summation of Fluids porosity measurement.

c. Samples are suitable for subsequent tests if no mercury penetration or adsorption occurs.

5.2.2.3 Limitations

Limitations of this method include:

a. Air trapped around the sample will yield erroneously high bulk volumes.

b. Vuggy samples or extremely high permeability samples will be penetrated by the mercury resulting in low bulk volume values and rendering the samples unsuitable for further testing (see 5.2.3).

c. In most mercury displacement pumps, the sample is immersed approximately 50 millimeters beneath the mercury. This mercury height induces a pressure of about 1 psi (6.9 kPa) to the top of the plug. This may make the bulk volumes systematically low because of 1) conformance to microscopic surface roughness or 2) penetration into large pores. The depth of immersion is minimized (3 to 7 millimeters) in the Archimedes Mercury Immersion method (see 5.2.1).

d. The method is unsuitable for unconsolidated samples that are mounted in lead sleeves because of lead-mercury amalgamation. Mercury may also be trapped between jacketing materials and the sample; consequently, this is not a recommended procedure for jacketed samples.

5.2.2.4 Accuracy

The measurement can be reproduced to 0.01 cm³ if the pump has been calibrated and is zeroed for each sample.

5.2.2.5 Apparatus

Figure 5-3 illustrates a high pressure, volumetric mercury displacement pump. The sample chamber will hold samples up to approximately 25 cm³ in volume.

5.2.2.6 Procedure/Calculations

The bulk volume of a regularly or irregularly shaped core sample is obtained by mercury displacement. The chamber, with no sample in place, is filled with mercury to the reference level. The instrument volume read-out is zeroed. The mercury level is then lowered, the sample is inserted and the chamber is again filled to the reference level. The instrument volume read-out is obtained. The difference between this reading and the instrument zero is the bulk volume of the sample.

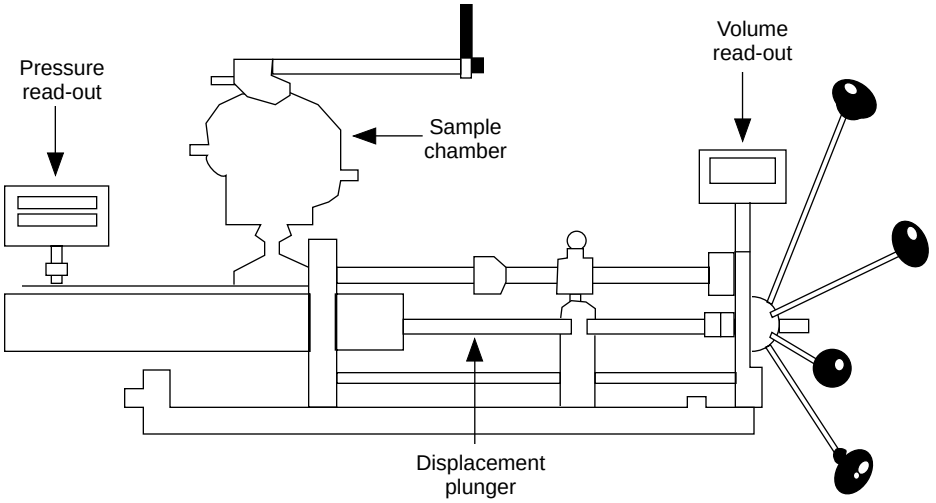


Figure 5-3—Volumetric Mercury Displacement Pump

5.2.2.7 Precautions

The mercury and chamber should be clean and free from surface films, solids, and oils.

5.2.2.8 Calibration

The mercury pump is calibrated with steel billets of known volume.

5.2.3 Caliper

5.2.3.1 Principle

Samples which are right cylinders or other regular shapes may be calipered to obtain bulk volume. A micrometer or vernier caliper, which can be read to the nearest 0.002 cm, can be used to measure length and diameter. A minimum of five measurements is recommended.

Note: Samples with surface vugs and/or open fractures may be analyzed using this approach if bulk volume data are calibrated on samples using the technique discussed in 5.2.3.6.

5.2.3.2 Advantages

Advantages of this method include:

a. The sample may be used for other tests.
b. The procedure is rapid.

5.2.3.3 Limitations

Limitations of this method include:

a. Samples with uneven shapes cannot be measured by this method.
b. Irregularities in sample surfaces may yield erroneously high bulk volumes.
c. Calipered bulk volumes are not generally recommended for porosity methods where $PV = (BV - GV)$. The accuracy of the bulk volume is not as critical where $f = PV / BV$ and the pore volume is determined by direct measurement (see 5.3.2.2). Where rock type (vuggy or fractured) dictates the use of the caliper technique, data should be calibrated as discussed in 5.2.3.6.
d. This method is not preferred for jacketed, poorly consolidated, or unconsolidated samples. If employed, corrections for screen and jacket thickness must be applied to both calipered length and diameter.

5.2.3.4 Accuracy

Length and diameter measurements of actual samples have been shown to be reproducible with a 99 percent confidence level (within 3 standard deviations) to within 0.15 millimeters for length and 0.04 millimeters for diameter. Bulk volumes can be repeated to 0.15 cm^3. See 5.2.3.6 for accuracy improvement.

5.2.3.5 Apparatus

Vernier or digital calipers may be utilized.

5.2.3.6 Procedure

The length and diameter of a cylinder or dimensions of a regularly shaped sample are measured at a minimum of five different positions to define any irregularities in the shape. Small deviations in the shape may be averaged out. Several samples without surface vugs and/or open fractures, whose gross dimensions cover the range of samples to be analyzed, should have their bulk volume determined by an appropriate immersion technique. Their bulk volume should then be determined using the exact caliper technique that will be used for measurement of subsequent samples. Correction factors may then be determined to calibrate calipered bulk volumes to those measured by immersion. These factors are then

applied to subsequent calipered bulk volume measurements to correct them to an equivalent immersion bulk volume.

Less time is required and more readings are taken if digital calipers are used. Compared to vernier calipers, the measurements are not as susceptible to human error since the readings are displayed as digital output. Digital calipers can be interfaced with a computer so the readings will be automatically recorded. About 10 length or diameter readings can be taken in less than 60 seconds.

5.2.3.7 Calculations

The cross-sectional area of the cylindrical sample is calculated from the average diameter and multiplied by the average length to obtain bulk volume. Appropriate formula can be used for noncylindrical samples.

5.2.3.8 Precautions

Precautions for this method include:

a. Diameter measurements should be taken at a minimum of five positions evenly spaced along sample length.
b. Length measurements should be obtained at a minimum of five positions around the sample's periphery.
c. Ensure that calipers are zeroed between successive measurements.
d. When measuring diameters and lengths of samples with surface irregularities, ensure that calipers do not intrude into surface vugs or fractures yielding measurements less than the true dimensions.

5.2.4 Archimedes (Buoyancy) With Fluids Other Than Mercury (e.g., Brine, Refined Oil, or Toluene)

5.2.4.1 Principle

A body placed in a liquid is buoyed up by a force equal to the weight of the displaced liquid.

5.2.4.2 Advantages

Advantages of this method include:

a. Accurate values can be attained if proper technique is used.
b. The sample can be totally saturated with liquid for other tests which may be desired.
c. If the sample is 100-percent saturated with a single fluid prior to the measurement of bulk volume, the pore volume, grain volume, and grain density can be calculated from recorded weights.

5.2.4.3 Limitations

Limitations of this method include:

a. The liquid may be unsuitable for subsequent tests and have to be removed.
b. Cores containing vugs should not be measured by this method.
c. Liquids which may leach the sample or cause swelling of the matrix cannot be used.
d. Vugs or large pores in extremely high permeability samples will be filled while submerged under the liquid, resulting in low bulk volume values (see 5.2.2.3, b.). Filling the vugs prior to measurement will allow a direct measurement of the bulk volume when the submerged weight is taken.
e. This is not a preferred technique for jacketed, poorly consolidated/unconsolidated samples because of the possibility of trapping extraneous fluid volume between the plug surface and the jacket.

5.2.4.4 Accuracy

The measurement of weight can be repeated within ±0.015 grams if air is not trapped when the sample is submerged and the temperature remains constant. The accuracy of bulk volume measurements will vary depending upon the density and volatility of the liquid used in the measurements. Good technique should yield repeatable bulk volume within 0.01 cm^3.

5.2.4.5 Apparatus

An analytical balance accurate to one milligram, fine wire cradle, liquid container, and thermometer are required. Figure 5-4 shows two possible experimental configurations.

5.2.4.6 Procedure

5.2.4.6.1 A sample is saturated with a liquid of known density, such as a nondamaging brine, light refined oil or a high boiling point solvent. The sample can be essentially 100-percent saturated with liquid by evacuating the pore space, introducing the saturant, and following with pressure saturation (see 5.3.2.2.3.6).

5.2.4.6.2 Excess liquid is carefully removed (avoiding grain loss) from the sample and the saturated sample is weighed in air. When removing excess liquid from the sample surface, precautions must be taken to ensure that fluids are not removed from pores exposed at the surface. Materials (such as dry towels) which remove liquid from surface pores due to capillary action should be avoided, as should any mechanical method such as violent shaking. Rolling the sample over a lint free cloth or paper towel dampened with the saturating liquid, or carefully wiping the sample with the dampened cloth or the fingers are acceptable methods for removing excess liquid.

5.2.4.6.3 A beaker is filled with the saturating liquid. A fine wire (maximum diameter of 1.0 millimeter) cradle attached to the stirrup of a balance is first submerged under the liquid to a reference mark, and the balance is tared. The sample is then placed on the cradle, submerged to the reference mark, and a submerged weight of the sample is obtained (see Figure 5-4A).

5.2.4.6.4 Figure 5-4B illustrates an alternate procedure. A beaker is filled with the saturating liquid and placed on a balance. A fine wire cradle is then lowered into the liquid to the reference mark, and the balance is tared. The saturated sample is then placed on the cradle, submerged to the reference mark, and the immersed weight of the sample is obtained.

5.2.4.6.5 A check on the pore volume can be made by taking (a) the difference in the weight of the 100-percent saturated sample in air and the dry weight, and (b) dividing by the saturating liquid density.

Note: It is not essential for the sample to be 100-percent saturated with the liquid for the determination of the bulk volume. The technique provides an accurate bulk volume as long as the sample is not imbibing liquid when the weight is taken while suspended under the liquid. However, if the sample is not 100-percent saturated, the pore volume, grain volume, and grain density calculated from the weights will be incorrect.

5.2.4.7 Calculations

Bulk volume from the apparatus shown in Figure 5-4A equals the initial weight of the saturated sample (or partially saturated sample with stabilized weight) in air minus the weight when submerged, divided by the density of the immersion liquid. Bulk volume from the apparatus shown in Figure 5-4B equals the immersed weight of the saturated sample divided by the density of the immersion fluid.

A

$$BV = \frac{\text{Saturated Weight} - \text{Immersed Weight}}{\text{Density of Immersion Fluid}}$$

B

$$BV = \frac{\text{Immersed Weight}}{\text{Density of Immersion Fluid}} \quad (6)$$

5.2.4.8 Precautions

Precautions for this method include:

a. Care must be taken in the design of the apparatus. The arrangement should be such that both the samples and the wire cradle are totally immersed during measurement, are not supported in any way by the immersion container, and only a length of straight wire (maximum diameter of 1.0 millimeter) penetrates the surface of the liquid. The wire and fluid should be clean.

b. It is essential to use the correct fluid density for the temperature of the liquid at the time of the submerged weight measurement. The density of the fluid should be known to at least the nearest 0.005 gm/cm^3. An error of 0.005 gm/cm^3 results in a porosity bias of 0.5 porosity points (porosity expressed as percent) and a grain density bias of 0.01 gm/cm^3. The density of the fluid can be measured with a pycnometer, an electronic digital density meter, various specific gravity balances, or it can be calculated using a silicon standard available from the National Institute of Standards and Technology.

$$V_{SS} = \frac{W_{SS}}{\rho_{SS}} \quad (7)$$

Where:

V_{SS} = silicon standard volume.
W_{SS} = air weight of silicon standard.
ρ_{SS} = silicon standard density from National Institute of Standards and Technology.

$$\rho_F = \frac{W_{SS} - W_{ISS}}{\rho_{SS}} \quad (8)$$

Where:

ρ_F = density of fluid in which silicon is immersed.
W_{ISS} = immersed weight of silicon standard.

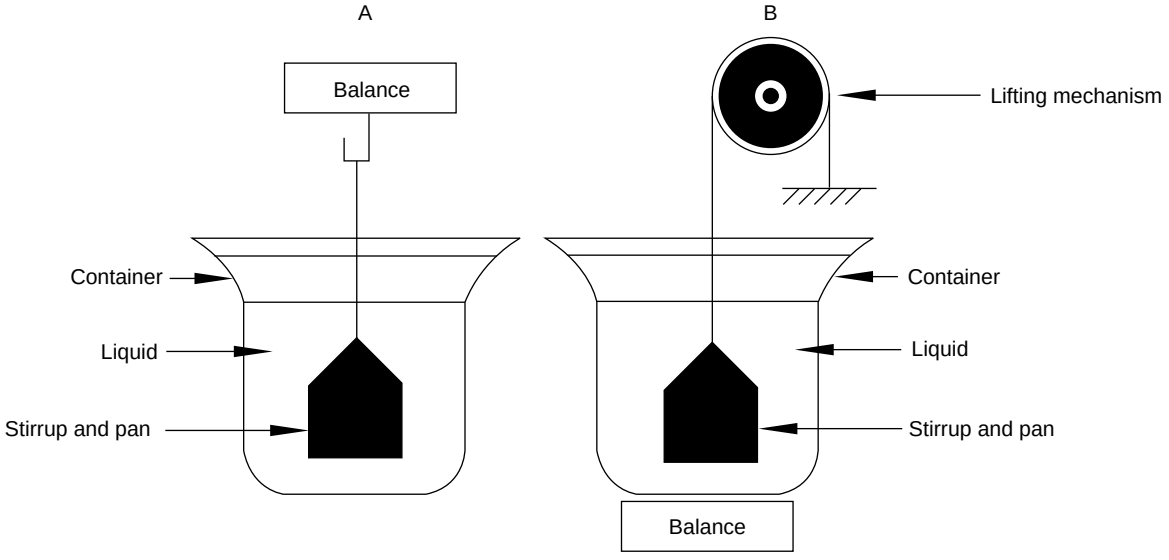


Figure 5-4—Archimedes (Buoyancy) With Fluids Other Than Mercury—Apparatus

c. It is probable that unstable decreasing weights indicate that fluid is imbibing into the sample, which will result in an erroneously low bulk volume.
d. Temperature fluctuations should be minimized as immersion liquid density will vary.
e. Care should be used in wiping the surfaces of the saturated sample so as to avoid grain loss or excess saturant removal. See 5.2.4.6.2 for suitable techniques.

5.2.5 Bulk Volume Calculated By Summing Direct Measurements of Grain Volume (5.3.2.1.1) and Pore Volume (5.3.2.2.1)

5.2.5.1 Calculations

The basic calculation is:

$$BV = GV + PV \quad (9)$$

5.3 PORE VOLUME MEASUREMENTS

Total pore volume is the total void space, both connected and isolated, in a rock sample.

5.3.1 Total Pore Volume From Disaggregated Sample Grain Density

The total pore volume equals the difference between the sample bulk volume and the disaggregated grain volume. Disaggregation is done to expose any isolated pore volume.

5.3.1.1 Dry Method for Grain Density/Grain Volume

5.3.1.1.1 Principle

The bulk volume of a cleaned sample is first determined (see 5.2). The sample is then dried, weighed, and disaggregated. After disaggregation, a weighed portion is placed into a Boyle's Law porosimeter to determine the grain volume (see 5.3.2.1.1). The grain volume of the total sample is calculated using the ratio of the dry weight of the consolidated sample to the dry weight of the disaggregated sample placed in the porosimeter.

5.3.1.1.2 Advantages

The advantages of this method are:

a. Fast.
b. Sample can be used for supplemental measurements requiring disaggregated sample. See Section 7, Supplementary Tests.

5.3.1.1.3 Limitations

The limitations of this method are:

a. Drying required.
b. Not viable for gypsum-bearing rock.
c. Not viable for rocks containing minerals such as halite that are soluble in the cleaning fluid.
d. Technique assumes any isolated pore volume remains isolated during the time the sample is dried.
e. If water fills the isolated pore space and is not removed while drying the sample, the calculated grain volume (*GV*) will be erroneously high.

5.3.1.1.4 Accuracy

Technique is capable of supplying grain density within ±0.01 g/cm^3. No comparative porosity data are available, but technique is expected to supply porosity within ±0.4 porosity units, or better.

5.3.1.1.5 Apparatus

The following apparatus are appropriate for this method:

a. Pulverizer with adjustable tolerance between the grinding plates.
b. U.S. Bureau of Standards 60-mesh sieve.
c. Analytical balance, with accuracy to 0.1 milligram.
d. Boyle's Law porosimeter modified to handle powdered samples. (1) Sample Cup cover (to keep the powdered sample in the cup).

5.3.1.1.6 Procedure

The procedure for this method is as follows:

a. Sample Preparation.
   1. Clean.
   2. Dry at 225°F (107°C).
   3. Cool in Desiccator.
   4. Weigh the sample and record weight (*W*).
   5. Repeat the above (steps 2 through 4) until weight (*W*) has stabilized.
   6. Determine Bulk Volume (*BV*) (see 5.2).
b. Crush the sample and pass the crushed sample through a 60-mesh sieve.
c. Dry a portion of the sample for Grain Volume measurement at 210° to 240°F (99° to 116°C) to a constant weight (W_d).
d. Determine the Grain Volume (GV_d) of the disaggregated sample portion by the Boyle's Law Double-Cell method (see 5.3.2.1.1).
   1. Calibrate the porosimeter with the sample cup cover in place.
   2. Place a known weight (W_d) of crushed and dried sample into the sample cup.
   3. Measure the Grain Volume of the disaggregated and dried sample portion with the cover in place.

5.3.1.1.7 Calculations

The calculations for this method are as follows:

$$PV = BV - GV \quad (10)$$

Where:

PV = total pore volume.
BV = bulk volume of original consolidated sample.
GV = grain volume of sample calculated by equation b.

$$GV = GV_d \times W/W_d \quad (11)$$

Where:

GV_d = measured grain volume of disaggregated sample portion placed in porosimeter.
W = original sample weight.
W_d = weight of dry, disaggregated sample portion placed in porosimeter cup.

$$GD = W_d/GV_d \quad (12)$$

Where:

GD = grain density of sample portion.

5.3.1.1.8 Precautions

Precautions for this method include:

a. The portion of rock selected for grain density measurement must be representative of the total sample.
b. See 5.3.2.1.1.8, Precautions for Boyle's Law Double-Cell (matrix cup) method for grain volume, items a through f.

5.3.1.1.9 Calibration

See 5.3.2.1.1.9, Calibration for Boyle's Law Double Cell (matrix cup) method for grain volume.

5.3.2 Effective Pore Volume of Aggregated Samples

Effective pore volume can be computed by subtracting the measured grain volume from the sample bulk volume, or by direct measurement of sample void volume.

5.3.2.1 Grain Volume Measurement

5.3.2.1.1 Boyle's Law Double-Cell (Matrix Cup) Method for Grain Volume

5.3.2.1.1.1 Principle

Boyle's Law: When the temperature remains constant, the volume of a given mass of ideal gas varies inversely with its absolute pressure.

$$\frac{V_1}{V_2} = \frac{P_2}{P_1} \quad \text{or} \quad P_1V_1 = P_2V_2 \quad (13)$$

Extension of the equation to account for temperature variation and nonideal gas behavior is required for accurate grain volume determination.

$$\frac{P_1V_1}{z_1T_1} = \frac{P_1V_1}{z_2T_2} \quad (14)$$

This latter equation should be utilized with a double-cell device when determining sample grain volume (GV). Gas is admitted into a reference cell of known volume (V_r) at a predetermined reference pressure (100 to 200 psig). The reference cell gas is then vented into a connected chamber of known volume containing a core sample. This results in a lower equilibrium pressure, from which GV is calculated. The GV is subsequently subtracted from bulk volume to yield pore volume and thence porosity.

5.3.2.1.1.2 Advantages

The advantages of this method include:

a. The test sample is not damaged in any way and can be used for other measurements.
b. The operation is quick, simple, and has excellent repeatability.
c. Irregular-shaped samples and vuggy cores can be tested.

5.3.2.1.1.3 Limitations

The limitations of this method include:

a. For good accuracy, extremely careful and frequent calibration is required.
b. Changes in either temperature or barometric pressure must be accounted for in the calculations.
c. The resultant porosity value will be higher than the true porosity value if gas adsorbs on the core surfaces. The use of helium gas minimizes this possibility.
d. This method is suitable for poorly consolidated/unconsolidated jacketed cores if proper techniques are employed. The volume of the jacketing material and end screens must be accurately determined and subtracted from the measured apparent grain volume. The jacketing material and screen solid volume can be determined by 1) direct measurement in a Boyle's Law Double-Cell matrix cup prior to use, or 2) jacketing material volume may be computed from a measured weight, and a previously-determined jacketing sleeve material density.

5.3.2.1.1.4 Accuracy

A well calibrated system will supply grain volume within ±0.2 percent of the true value. This corresponds to approximately ±0.03 cm^3 in one-inch diameter samples approximately 1 inch long and ±0.1 cm^3 in $1^1/_2$-inch diameter samples approximately 2 inches long.

5.3.2.1.1.5 Apparatus

The grain volume is measured in an apparatus consisting of two connected chambers of known volumes. An example of such an apparatus is shown in Figure 5-5.

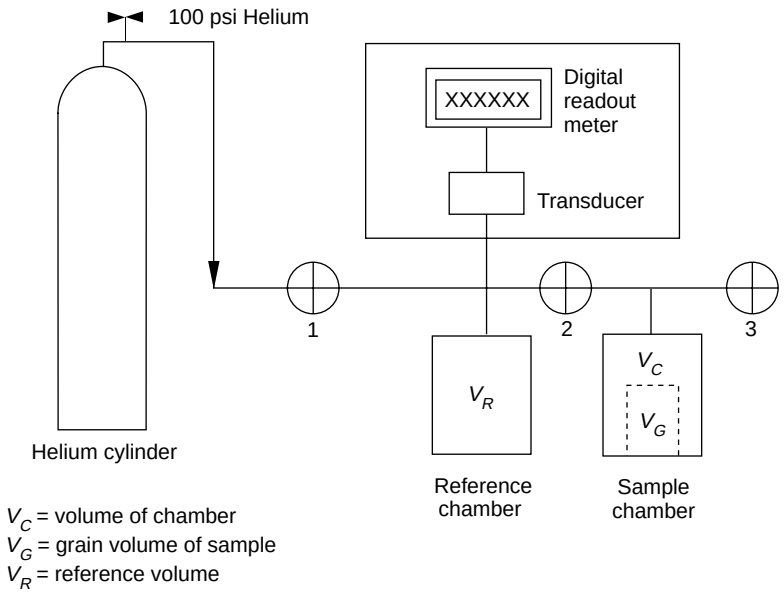


Figure 5-5—Double-Cell Boyle's Law Porosimeter

Note: For accurate measurements, it is important to (a) incorporate a valve having a zero displacement volume (e.g., a ball valve) between the reference volume and the sample chamber, or (b) the valve displacement volume must be known. The ball valve must always be vented to atmospheric pressure before it is turned to the closed position. Used in this manner, the valve bore volume is incorporated into the calibrated chamber volume and measurements are accurate. Otherwise, the valve displacement volume must be measured and accounted for in calculations for accurate measurement of grain volume.

5.3.2.1.1.6 Procedure

The porosimeter is first calibrated, yielding the reference chamber volume (V_r) and the sample chamber volume (V_c). A core plug is then placed in the sample chamber. Helium gas is admitted into the reference chamber at a predetermined pressure, typically 100 to 200 psig (690 to 1380 kPa). About 30 seconds should be allowed for pressure equilibrium and then p_1 (pressure indicated by the transducer digital readout) should be recorded. The gas is then allowed to expand into the sample chamber. The resulting lower pressure (p_2) is measured after the system has reached equilibrium (see 5.3.2.1.1.8, e). The grain volume of the sample is calculated using the gas law equations discussed in 5.3.2.1.1.7.

5.3.2.1.1.7 Calculations

The grain volume of the sample is calculated from the initial reference chamber pressure and the final system pressure by means of the gas law equation. The pore volume is the difference between the bulk volume and the grain volume.

The following grain volume equation is derived by a mass balance of gas within the reference and sample chamber:

$$\frac{P_1 V_r}{z_1 T_{1r}} + \frac{P_a (V_c - V_g)}{z_a T_{1c}} = \frac{P_2 V_r}{z_2 T_{2r}} + \frac{P_2 (V_c - V_g + V_v)}{z_2 T_{2c}} \qquad (15)$$

Where:

P_1 = absolute initial reference volume pressure.
P_2 = absolute expanded pressure.
P_a = absolute atmospheric pressure initially in sample chamber.
z_1 = z-factor of gas at P_1 and T_1.
z_2 = z-factor of gas at P_2 and T_2.
z_a = z-factor of gas at T_1 and atmospheric pressure.
T_{1r} = absolute temperature of reference volume at P_1.
T_{1c} = absolute temperature of sample chamber at P_1.
T_{2r} = absolute temperature of reference volume after P_2. is stabilized.
T_{2c} = absolute temperature of sample chamber after P_2 is stabilized.
V_g = grain volume.
V_c = sample chamber volume.
V_r = reference chamber volume.
V_v = valve displacement volume (from closed to open position).
p_1 = initial reference volume gauge pressure.
p_2 = final system gauge pressure.

If isothermal conditions exist ($T_1 = T_2$) and z values are assumed to equal 1.0, the equation reduces to:

$$V_g = V_c - V_r\left(\frac{P_1 - P_2}{P_2 - P_a}\right) + V_v\left(\frac{P_2}{P_2 - P_a}\right) \qquad (16)$$

If absolute pressures P_1 and P_2 are expressed as equivalent gauge pressures (i.e., $P_1 = (p_1 + P_a)$), and are substituted in the above equation, the following results:

$$V_g = V_c - V_r\left(\frac{p_1}{p_2} - 1\right) + V_v\left(1 + \frac{p_a}{p_2}\right) \quad (17)$$

If a zero displacement volume ball valve is used and the valve is always vented to the atmosphere before closing, V_v is included in the sample chamber volume, V_v = 0.0 and the equation further simplifies to:

$$V_g = V_c - V_r\left(\frac{p_1}{p_2} - 1\right) \quad (18)$$

Note: Calculations ignoring either *z* factors, or valve volume as small as 0.1 cm^3, can result in porosity errors of approximately 0.5 porosity points.

5.3.2.1.1.8 Precautions

Precautions for this method include:

a. The matrix cup must be designed to ensure that it will have the same internal volume each time it is reassembled.
b. The system must be recalibrated if there are changes in temperature or barometric pressure.
c. The core plug must be thoroughly and properly dried. Some samples require special drying techniques.
d. When pore water salinities (either from formation water or mud filtrate) are greater than 100,000 mg/l, samples should be leached with methanol to remove salts. Ignoring the leaching step when pore water salinities exceed 100,000 mg/l can bias porosities low by 0.4 porosity points (typical 20 percent porosity and a water saturation of 50 percent pore space).
e. Equilibrium pressure is essential for accurate grain volumes. Equilibrium is usually attained in 1 to 2 minutes, but low permeability and porosity cores require longer stabilization times (30 minutes to several hours). Longer times yield greater chance for temperature and barometric pressure changes.
f. Extraneous or dead volume (volume not occupied by the sample in the sample chamber) must be kept to a minimum or erroneous grain volumes will be determined. Solid stainless steel cylinders of known volume can be added to fill the sample chamber void volume left by shorter samples. When added, these steel volumes must be subtracted from the calculated grain volume to yield sample grain volume.

Note: If steel cylinders are present in the matrix cup during the calibration to yield V_c and V_r, their volumes should not be subtracted.

g. The pore volume of the sample to be measured should approximate the reference cell volume for best analytical results. Accuracy of the method on small, irregular test samples can be checked by determination of grain density on larger, and then smaller, similarly sized samples of a known grain density (i.e., Ottawa Sand).

5.3.2.1.1.9 Calibration

Porosimeter calibration varies with the instrument. In principle, two or more measurements are made; one is with the sample chamber filled with solid stainless steel cylinders of known volume. Subsequent measurements are made after removing one or more cylinders representing approximately 80, 60, 40, and 20 percent of the sample volume chamber. For best accuracy, sufficient cylinders should be removed to reduce p_1 by one-half.

Helium gas is admitted into the reference chamber at a predetermined pressure, typically 100 to 200 psig (690 to 1,380 kPa). About 30 seconds should be allowed for pressure equilibrium, and then p_1 (reference cell pressure indicated on the transducer digital read-out) is recorded. The gas is then expanded into the sample chamber.

The resulting lower pressure (p_2) is measured after the system has reached equilibrium (approximately 30 seconds). With valve volume (V_v) zero, temperature constant, and V_g known from volume of the steel cylinder in the chamber, V_r and V_c can be calculated from simultaneous solution of the appropriate equation presented in 5.3.2.1.1.7.

When V_v is not equal to zero or temperature varies, the solution becomes more complex, but the principle remains the same.

Note 1: In some systems equations are rearranged, and data are fit by linear regression over the cylinder volume range to yield V_c and V_r.

Note 2: Pressure transducers should be calibrated over 5 psi intervals with a dead weight tester or 0.02 percent full scale (or better) secondary standard pressure transducer. Transducer output can be fit to "true pressure" with a fourth degree polynomial using regression techniques. Check of the calibration can be made by measuring the volume of each steel cylinder and comparing it to the known volume. Agreement should be equal to or less than 0.03 cm^3 to assure porosity within 0.5 porosity points over the range of grain volumes measured.

5.3.2.2 Void Volume Measurement

5.3.2.2.1 Boyle's Law Single Cell Method for Direct Pore Volume

5.3.2.2.1.1 Low Confining Stress

5.3.2.2.1.1.1 Principle

Pore volume is determined in an apparatus consisting of a gas charged reference cell of known volume and initial pressure, which is then vented into a sample's pore volume. The sample is held in a core holder which utilizes an elastomer sleeve and end plugs. These conform closely to the sample when confining pressure is exerted on their external surfaces. The sleeve and end stems in turn exert compressive stress on the core sample. Pore volume is therefore determined directly using Boyle's Law. This is in contrast to the double cell method whereby grain volume is determined and pore volume is calculated by subtracting grain volume from bulk volume.

The core holder may be a Hassler, isostatic, biaxial, or triaxial load cell. Low confining stresses, which are generally 400 psig or less, are employed. In consolidated, hard rocks, the type core holder is of little significance as minimum pore space reduction occurs at low stress. Although these are typi-

cally referred to as "ambient measurements," it is important to note that some finite confining stress is necessary, and stress can have significant effects on unconsolidated or friable rocks.

Helium gas is admitted into the reference cell of known volume (V_r), at a predetermined reference pressure (100 to 200 psi). The reference cell gas is then vented into the sample pore volume. This results in a lower equilibrium pressure, from which the pore volume is calculated.

5.3.2.2.1.1.2 Advantages

Advantages of this method include:

a. The sample, if clean and dry at test initiation, is clean at test conclusion and ready for subsequent measurements.
b. Porosity and permeability can be run in sequence with a single loading of the sample.
c. Operation is quick and simple.
d. Harmful reactions between the rock and saturating fluid are eliminated by use of non-reactive gas.
e. Direct measurement of the pore volume eliminates the sensitivity of the pore volume to errors in the measurement of either *BV* or *GV*, where both *BV* and *GV* are large numbers relative to the pore volume, and $PV = BV - GV$.

5.3.2.2.1.1.3 Limitations

Limitations of this method include:

a. The system must be carefully calibrated for dead volume.
b. Changes in temperature and barometric pressure must be accounted for in the calculations.
c. The sample must be a good quality right cylinder with no surface vugs or chipped corners. (Vugs will yield erroneously low pore volume and non-parallel ends will yield high pore volume.) A porosity error exceeding 1.5 porosity units can result from a single, non-parallel sample face, which results in a reduction of sample length of 1.0 millimeter on one side of an otherwise one-inch diameter by one-inch long right cylindrical plug.
d. The sample must be clean of hydrocarbons and dry, or erroneously low pore volume will be determined.
e. The gas used must not adsorb to active mineral sites such as clays or carbonaceous materials. Adsorption results in erroneously high pore volume, and can be minimized by the use of helium gas.
f. Some low permeability rock (<0.01 md) may require a long time (thirty minutes to several hours) to reach pressure equilibrium. Longer times yield greater chances for temperature and barometric pressure changes.
g. When pore water salinities (either from formation water or mud filtrate) are greater than 100,000 mg/l, samples should be leached with methanol to remove salts. Ignoring the leaching step when pore water salinities exceed 100,000 mg/l can bias porosities low by 0.4 porosity points (typical 20 percent porosity and a water saturation of 50 percent pore space).
h. This method is suitable for jacketed, poorly consolidated/unconsolidated samples if proper precautions are observed. The void volume of the screens or porous plates attached to the plug faces must be determined. This volume must be subtracted from the jacketed sample's measured void volume to yield sample pore volume. A solid non-porous jacketed plug equal to sample diameter may be prepared and tested as a sample. This will yield the screen or porous plate void volume, as well as any volume resulting from overlap of the jacketing material on the sample ends.
i. Porosity is determined at low confining stress, which results in a porosity higher than present in the reservoir.

5.3.2.2.1.1.4 Apparatus

The basic apparatus shown in Figure 5-6 is the same as that illustrated for the double-cell Boyle's Law porosimeter shown in Figure 5-5 (see 5.3.2.1.1.5). The primary difference is the design of the sample chamber, which eliminates volume around the periphery of the sample.

5.3.2.2.1.1.5 Procedures

The porosimeter is first calibrated, yielding the reference chamber volume (V_r) and system dead volume (V_d). A clean, dry core plug is then inserted into an elastomer sleeve. An end stem with diameter equal to that of the plug is placed in contact with each end of the sample. A confining stress of 400 psi or less is applied to the external surface of the elastomer. If the sample is confined in an isostatic core holder, an equal

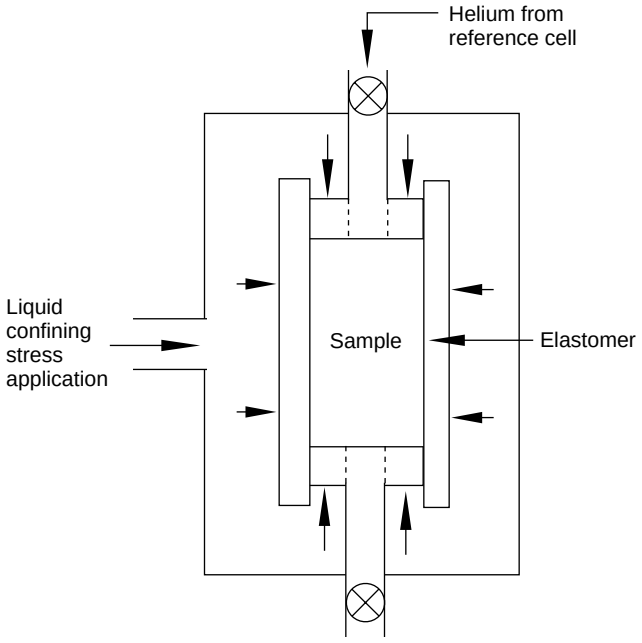


Figure 5-6—Schematic of Isostatic Load Cell for Direct Pore Volume Determination

confining stress is also applied to the outside surface of the end stems (see Figure 5-6).

Helium gas is admitted to the porosimeter reference chamber (V_r) at a predetermined pressure (p_1), typically equal to 100 to 200 psig (690 to 1380 kPa). Pressure is recorded, and the reference chamber is vented into the void volume of the sample. The resulting lower equilibrium pressure (p_2) is recorded. Pore volume of the sample is computed utilizing equations in 5.3.2.2.1.1.6.

5.3.2.2.1.1.6 Calculations

Sample volume is determined by expanding helium from a reference cell at known initial pressure directly into the porous rock. The following pore volume equation is derived by mass balance of gas within the reference cell, system dead volume, valve volume and sample pore volume.

$$\frac{P_1 V_r}{Z_1 T_{1r}} + \frac{P_a(V_p + V_d)}{Z_a T_1} = \frac{P_2(V_r + V_p + V_d + V_v)}{Z_2 T_2} \qquad (19)$$

Where:

P_1 = absolute initial reference volume pressure.
P_2 = absolute expanded pressure.
P_a = absolute atmospheric pressure initially in sample.
Z_1 = gas deviation factor at P_1 and T_1.
Z_2 = gas deviation factor at P_2 and T_2.
Z_a = gas deviation factor at P_a and T_1.
T_{1r} = absolute temperature of reference volume at P_1.
T_1 = absolute temperature of sample pore volume at P_a.
T_2 = absolute temperature of reference volume and sample after P_2 is stabilized.
V_r = reference chamber volume.
V_p = sample pore volume.
V_v = valve displacement volume (from closed to open position).
V_d = system dead volume.

If isothermal conditions exist, ($T_1 = T_2 = T_{1r} = T_1$) and:

$$\frac{P_1 V_r}{Z_1} + \frac{P_a(V_p + V_d)}{Z_a} = \frac{P_2(V_r + V_p + V_d + V_v)}{Z_2} \qquad (20)$$

grouping terms yields:

$$V_p = \frac{V_r\left(\frac{P_1 Z_2}{P_2 Z_1} - 1\right) - V_v}{1 - \frac{P_a Z_2}{P_2 Z_a}} - V_d \qquad (21)$$

Note: This equation is valid if no change in pore volume occurs as the sample pore pressure increases from P_a to P_2, i.e., (a) when the net effective confining stress remains constant, or (b) when it is not constant, but pore volume change with net confining stress change is negligible.

5.3.2.2.1.1.7 Precautions

Precautions for this method include:

a. Confining pressure on the external surface of the elastomer sleeve should be applied by liquid and not gas. Gas may diffuse into and through the sleeve with extended testing times.
b. Durometer of the elastomer sleeve must be low enough (suggest 50 durometer maximum) so that the sleeve seals on the metal end stems and properly conforms to the sample surface at the low confining stress utilized.
c. Samples with ends that deviate slightly from parallel can sometimes be successfully tested by utilization of two deformable, rubber discs. Each disc should be soft, equal to end butt diameter, and contain a drilled center hole. A single disc should be inserted between each metal end stem and the sample face. The disc will compress and conform to fill the void space between sample and end stem.

Note: This is recommended only when sample ends cannot be squared. The procedure is not a substitute for good quality samples.

5.3.2.2.1.1.8 Calibration

Equipment calibration utilizes (a) billets of known volume which are added or removed from a special chamber introduced only for calibration, or (b) a series of steel plugs precision bored to yield a variety of void volumes that cover the range of pore volumes to be measured. The reference volume is charged to initial pressure and vented into the special chamber or into the known void volume of the confined calibration plug. The measurement is repeated with multiple calibration billets or plugs. The pore volume equations can be solved simultaneously to yield the reference cell volume V_r and the dead volume. These values then become a constant for subsequent measurements of pore volume.

Dead volume (V_d) of the system can also be determined by utilizing a solid steel billet as a sample within the core holder or by placing the end stems adjacent in the elastomer sleeve and applying the same low confining stress to be used for sample measurement. The charged reference cell (V_r) is vented to the steel billet or to the adjacent end stems, after which the pore volume equation is used to calculate V_d. This value also remains constant as long as no changes are made to the system. Steel check plugs of known void volume can be measured as a final check of system operation and proper V_r and V_d values. These check plugs should also be utilized as daily confirmation of proper equipment performance.

5.3.2.2.1.1.9 Accuracy/Precision

A properly calibrated system will yield pore volumes within ±0.03 cm^3 in perfect cylinders. Measurements on actual core samples indicate scatter of approximately 0.1 cm^3 on samples of 50 cm^3 bulk volume, yielding porosity deviation of ±0.2 porosity units from the true value.

5.3.2.2.1.2 Elevated Confining Stress

5.3.2.2.1.2.1 Principle

The principle is similar to that discussed under low confining stress; however, in this measurement, porosity is determined at elevated stress. These laboratory data in turn are used to estimate reservoir porosity at in-situ reservoir stress conditions. Unlike low confining stress measurements, knowledge of the (a) elevated confining stress magnitude, and (b) method of application (isostatic stress, triaxial stress, biaxial stress, or uniaxial strain) must be known to scale results to reservoir conditions (see Figures 5-7 and 5-8). Historically, most routine pore volume measurements at elevated stress have been made with an isostatic (equal in all directions) confining stress, because this is an easily applied, relatively inexpensive method. Data are subsequently presented as porosity versus effective (confining pressure minus pore pressure) isostatic stress. In some reservoirs, a more complex and time consuming pore volume compressibility should be measured to more accurately determine porosity under reservoir conditions. These compressibility measurements typically require that liquid be present in the pore space to model liquid-rock interaction effects, if any,[1] and are beyond the scope of this document (see references in 5.6).

5.3.2.2.1.2.2 Advantages

Advantages of this method include:

a. Advantages are similar to those for low confining stress (see 5.3.2.2.1.1.2).
b. Measurements at elevated confining stress more closely represent original reservoir porosity than measurements at zero or low confining stress.

5.3.2.2.1.2.3 Limitations

Limitations of this method include:

a. Limitations are similar to items a through h for low confining stress (see 5.3.2.2.1.1.3).
b. Penetration of the elastomer sleeve into the depressions between sand grains or surface vugs on the sample surface as the confining stress increases may indicate pore volume reduction, even if no sample compression actually occurs. This penetration seldom exceeds 0.2 porosity units for non-vuggy sandstones (e.g., 10.0 percent to 9.8 percent), and while present, normally has no significant impact on porosity values. For vugular samples, this error can be significant. To reduce this error, a mylar or metal inner sleeve may be used. Surface effects can be minimized by increasing sample radius (r), as the ratio of surface area to bulk volume equals $2/r$ and decreases as sample radius increases.
c. The initial confining stress must be high enough to seal the elastomer sleeve to the sample and sample holder end butts, and to press the sample holder end butts tightly against the sample face. Sealing of the elastomer to the sample surface is dependent on the elastomer stiffness and sample roughness. Sealing stress must also be high enough to prevent bypassing of gas if permeability is to be measured in conjunction with porosity. Consequently, two opposing effects on porosity can occur. Poor conformance of the sample holder end butts with the sample, which yields porosity values that are too high, may be offset by sample pore volume compression. Use of good quality, right cylindrical samples with parallel ends minimizes conformance difficulties.
d. The resultant porosity at any elevated stress may be dependent on the rate of stress application, on the stabilization time and on the stress history.[2]
e. Reservoir effective stress must be known or estimated in order to properly utilize the laboratory-determined elevated-stress porosity data. Actual reservoir stresses causing pore volume reduction are usually not isostatic. The maximum principal stress in most reservoirs is vertical. This vertical effective stress equals the weight per unit area of overburden rock minus the reservoir pressure. Overburden stress can be computed from the depth and a downhole density log, or estimated assuming rock density equals 1.0 psi/ft.[3] Reservoir pressure can be measured, or in normally pressured areas, is assumed to be depth times the water gradient, which is approximately 0.45 to 0.5 psi/ft.

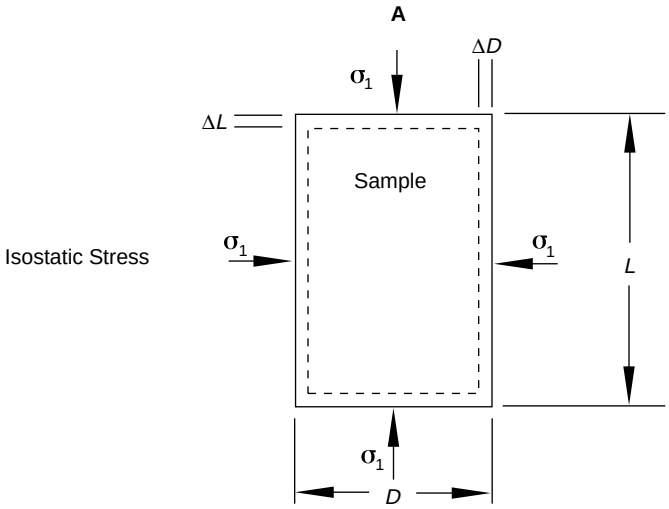
Note: Offshore reservoirs have a component of overburden stress resulting from water depth and density, as well as overburden rock.

Typically, horizontal stresses are some fraction of the vertical stress, and can be estimated from hydraulic fracture treatments or formation integrity (leakoff) tests.[4,5] The mean reservoir effective stress is an average of vertical and horizontal effective stresses. Consequently, the mean reservoir effective stress is typically less than an isostatic stress equal to the vertical effective stress.

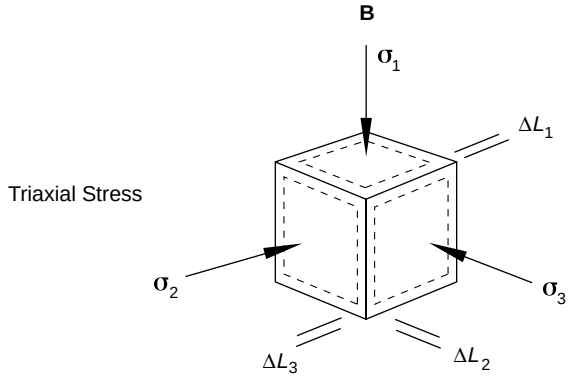
f. The presence of water within the pore space has been shown to weaken some rocks, thereby increasing pore volume reduction as confining stress is applied.[1] Since routine measurements, as discussed here, are on cleaned and dried rock samples, while the reservoir contains interstitial water, some uncertainty is introduced. While this effect is minimal in hard, well-cemented rocks, the importance of this effect can be quantified by conducting specialized tests on samples that contain water.
g. Friable or poorly-consolidated cores can exhibit significant compression at low confinement stresses.

5.3.2.2.1.2.4 Apparatus

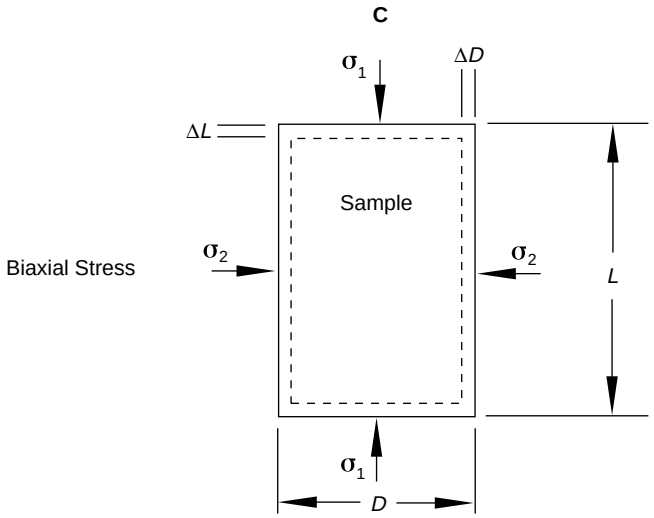
The apparatus is similar to that for low confining stress. However, high confining stress measurements require a sleeve material made of a tough elastomer that can withstand forces imposed during confining stress application, and yet will conform to the sample at low stress.



Under isostatic stress loading, equal stress is applied to the sample in all directions, and sample strain can occur on all axes. Excessive porosity reduction typically occurs when the imposed isostatic stress is equal to the vertical reservoir stress (i.e., the overburden stress).

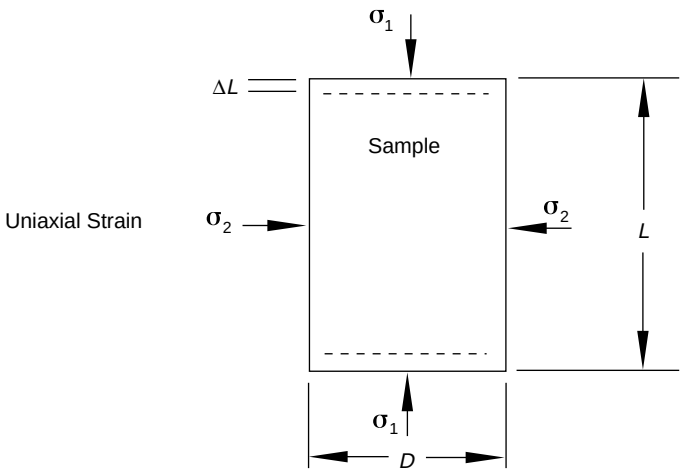


Under true triaxial stress conditions, unequal stress is applied to the three major axes of the sample. In the general case, strains will be different on each axis. Typically, a cube or rectangular prism-shaped sample will be used.



Biaxial stress loading conditions are a special case of triaxial stress loading. In the biaxial stress loading of a cylinder, the stress parallel to the cylinder axis is different than the stress applied around the sample's circumference. Strains can occur parallel to both the axis and diameter of the cylinder.

Figure 5-7—Laboratory Loading Schematic



Uniaxial strain compression is a special case of biaxial stress loading; the stress applied to the circumference is just sufficient to maintain the diameter constant as the stress parallel to the cylinder axis is increased. Strain occurs only parallel to the axis of the cylinder.

Figure 5-8—Laboratory and Reservoir Loading Schematic

5.3.2.2.1.2.5 Procedures

Procedures for elevated confining stress measurements are similar to those for low confining stress discussed in 5.3.2.2.1.1.5.

Typically, sample pore volume is determined at selected levels of increasing confining stress. Equilibrium pressure within the sample pore space at each confining stress is normally vented prior to subsequent pore volume measurement. Equations shown in 5.3.2.2.1.1.6 are used to compute pore volumes.

5.3.2.2.1.2.6 Calculations

The basic equations for pore volume (*PV*) calculation are the same as for low confining stress measurements, but calculation of porosity must also account for reduction in sample bulk volume (*BV*) as confining stress increases. Approaches that can be used include the following:

$$\text{Porosity} = PV/(\text{Non-stressed } BV - \Delta PV) \quad (22)$$

Where ΔPV = reduction in pore volume between initial *PV* and *PV* at any stress. This assumes the bulk volume reduction at any stress equals the pore volume reduction at the same stress.

$$\text{Porosity} = PV/(GV + PV) \quad (23)$$

Where *PV* = pore volume at any stress. This assumes grain volume (*GV*) as determined at zero stress is constant at all applied confining stress levels.

$$\text{Porosity} = PV/\text{calculated } BV \quad (24)$$

Measure pore volume (*PV*), sample diameter, and length at each stress imposed and calculate bulk volume. This is not routinely done.

5.3.2.2.1.2.7 Calibration

Calibration of equipment utilizes the same principles discussed in low confining stress (see 5.3.2.2.1.1.8). Validity of the system operation at elevated confining stress can be confirmed by measurement of steel check plug void volumes at the confining stresses selected for sample pore volume determination.

5.3.2.2.1.2.8 Accuracy/Precision

A properly calibrated system will yield pore volume accuracy of the same magnitude as observed at low confining stress (see 5.3.2.2.1.1.9), yielding porosity scatter of ±0.2 to 0.3 porosity units.

5.3.2.2.2 Summation of Fluids Porosity Summary

The "Summation of Fluids" technique of porosity determination is discussed in detail in the Fluid Saturation Methods under "Retort Method at Atmospheric Pressure" (see 4.2). It furnishes porosity data quickly as samples do not require cleaning or drying. The pore volume is computed by measuring and summing oil, gas, and water volumes present in a freshly recovered core sample. Porosity is determined by dividing pore volume by bulk volume of the rock sample.

When proper procedures are followed, and rocks are reasonably uniform and contain no hydratable minerals to complicate determination of water volumes, Boyle's Law porosities on adjacent core will typically agree within ±0.5 porosity units.[6] Without special precautions and techniques, the summation of fluids porosity can be erroneously high due to excess water recovery from hydratable minerals. This technique is not valid on weathered core from which liquids have evaporated. Measurements on weathered rock yield an erroneously low gas volume, and hence porosity, because the

injected mercury at the pressure utilized does not fill the smaller pores from which water has evaporated.

Grain density can be calculated from the measured volumes of rock and fluids. This can be compared to an estimated grain density based on lithology. The magnitude of the difference between calculated and estimated values yields a quality control check that will expose erroneous measurements or data calculation. This is not valid when extraneous minerals (heavy or light) are present in the rock. These conditions make the grain density variable and difficult to accurately estimate.

Retorting of the rock destroys the sample, so adjacent core must be used for permeability measurements. These permeability samples can be tested for porosity utilizing the Boyle's Law procedure, and compared with the summation of fluids values as a quality control check. Perfect sample-to-sample agreement is not expected, but the data should exhibit no bias.

5.3.2.2.3 Liquid Saturation Method

5.3.2.2.3.1 Principle

The measurement of porosity (connected pore space) by the liquid saturation method involves the gravimetric determination of pore volume by obtaining: (a) the weight of the core sample clean and dry, (b) the weight of the sample saturated with a liquid of known density, and (c) the weight of the saturated sample submerged in the same liquid.

5.3.2.2.3.2 Advantages

Advantages of this method include:

a. Many samples can be handled at one time.

b. Provides a direct measurement of pore volume.

c. Refer to 5.2.4.2, a and b.

5.3.2.2.3.3 Limitations

Limitations of this method include:

a. The procedure is slow in regard to total elapsed time of measurement.

b. The procedure is basically accurate, but limited to samples which can be 100-percent saturated and the saturated weight successfully determined.

c. Cores containing surface vugs are not recommended for porosity determination by this method because of the potential loss of liquid from the vugs during the weighing process. If liquid is lost, this volume would not be included in the pore volume, and erroneously low porosity will be calculated. However, grain volume can be accurately determined on vuggy cores by this method.

d. Refer to 5.2.4.3, a and c.

5.3.2.2.3.4 Accuracy

The method should determine porosity (connected pore space) within 0.5 porosity units and grain volume to ±0.2 percent of true value if proper technique is used.

5.3.2.2.3.5 Apparatus

Appropriate apparatus for this method include:

a. Analytical balance accurate to 1 milligram.
b. Suitable containers which can be used to hold deaerated liquid under vacuum.
c. Vacuum desiccator and pressure saturator.
d. A suitable vacuum source capable of maintaining less than 0.1 millimeter of mercury pressure.
e. A filtered, low-viscosity, low vapor-pressure liquid of known density for the purpose of saturating the core samples. Some liquids which have been used are: 1) brines, 2) refined laboratory oils, 3) decane, and 4) toluene.
f. Suitable containers which can be used to hold saturated cores under deaerated liquid.

5.3.2.2.3.6 Procedure

Obtain the weight of the dry, clean sample. The sample should be desiccated over a suitable dehydrating material, such as $CaCl_2$ or silica gel, prior to the determination of the dry weight.

Place the weighed, dry sample in a chamber (vacuum desiccator and pressure saturator) and apply a high vacuum for about 8 hours. The evacuation period may be followed by injection of CO_2 to remove the absorbed air on the rock. This replaces adsorbed gases such as O_2 or N_2 with CO_2, usually a more soluble gas in liquids typically used. Several cycles of evacuation and CO_2 injection may be required for low permeability samples. Longer CO_2 contact time and longer vacuum cycles are required to displace N_2 in the tighter rock. For very low-permeability samples, the evacuation period should be as long as 12 to 18 hours (overnight).

The liquid used to saturate the cleaned core sample should be free from dissolved air; therefore, the liquid should be deaerated before introduction into the core. Extreme care should be taken to avoid air contact with the liquid after its deaeration. Hydrocarbons are stronger wetting fluids than brines and are preferred if only porosity measurements are desired and samples saturated with brine are not required for further testing.

The deaerated liquid is drawn into the evacuated vessel containing the core sample. The core is allowed to saturate. After the sample has been completely submerged in the liquid, the vacuum is continued for an additional 30 minutes to 1 hour.

The preferred method is to then pressure the liquid surrounding the sample to 2,000 to 3,000 psi (13,800 to 20,700 kPa) for at least four hours to assure complete saturation. Pressure saturation should result in the liquid entering addi-

tional voids which are not well connected or where spontaneous imbibition has not occurred. The sample is then removed from the saturating vessel and weighed submerged in the saturating liquid.

Excess liquid is carefully removed (avoiding grain loss) from the sample and the saturated sample is weighed in air. When removing excess liquid from the sample surface, precautions must be taken to ensure fluids are not removed from pores exposed at the surface. Materials (such as dry towels) which remove liquid from surface pores due to capillary action should be avoided, as should any mechanical method such as violent shaking. Rolling the sample over a lint free cloth or paper towel dampened with the saturating liquid, or carefully wiping the sample with the dampened cloth or the fingers are suitable techniques for removing excess liquid.

After weighing each batch of samples, the density of the deaerated saturant used in the saturation chamber is determined. This must be done with accuracy, since the saturant density is probably the greatest source of error unless Equation 5.3.2.2.3.8e is used. Refer to procedures described in 5.2.4.6 for the determination of bulk volume. When coupled with the original dry weight and fluid density, grain volume is computed directly (see Equation 5.3.2.2.3.8c).

For storage until use, the core plugs are usually transferred to screw-capped jars or other suitable containers that are filled with deaerated liquid in order to minimize the presence of air in the container.

5.3.2.2.3.7 Precautions

Precautions for this method include:

a. Special precautions are necessary to ensure complete saturation. Low permeability cores may not be fully saturated by this method.
b. For acceptable accuracy, it is essential to use the correct fluid density at the time the saturated weight of the sample is determined.
c. Error in the pore volume can result from improper wiping of the sample prior to the determination of the saturated weight.

5.3.2.2.3.8 Calculations

The calculations for this method are as follows:

a. $$PV = \frac{\text{Saturated Weight} - \text{Dry Weight}}{\text{Density of Saturant}}$$

b. $$BV = \frac{\text{Saturated Weight} - \text{Immersed Weight}}{\text{Density of Immersion Liquid}}$$ (see 5.2.4)

c. $$GV = \frac{\text{Dry Weight} - \text{Immersed Weight}}{\text{Density of Immersion Liquid}}$$

d. Pore Volume check: $PV = BV - GV$ (PV by difference should agree with PV calculated in a.)

e. $$\text{Porosity} = \frac{PV}{BV} = \frac{\dfrac{\text{Saturated Weight} - \text{Dry Weight}}{\text{Density of Saturant}}}{\dfrac{\text{Saturated Weight} - \text{Immersed Weight}}{\text{Density of Immersion Liquid}}}$$

If temperature is constant, and density of saturant and immersion liquid are equal, fluid density cancels and is not needed in the above equation to calculate porosity.

$$\text{Porosity} = \frac{\text{Saturated Weight} - \text{Dry Weight}}{\text{Saturated Weight} - \text{Immersed Weight}}$$

f. Porosity check: $$\text{Porosity} = \frac{BV - GV}{BV}$$

(Porosity should agree with that calculated in e.)

g. $$\text{Grain Density} = \frac{\text{Dry Weight}}{GV}$$

Where:

PV = pore volume.
GV = grain volume.
BV = bulk volume.

5.4 HISTORICAL PROCEDURES

This section lists and summarizes techniques that have been used historically to measure porosity, bulk volume, grain volume, and grain density. These have generally been replaced by newer techniques. Details about these procedures can be found in the First Edition of API RP 40, *API Recommended Practice for Core-Analysis Procedure,* August 1960.

5.4.1 Mercury Displacement

This technique consists of immersing a dried core sample in mercury inside a calibrated pycnometer. The volume of mercury displaced by the sample is weighed to determine the bulk volume of the core sample. (API RP 40, 1960, Sec. 3.311, p. 15; Sec. 3.56, p. 26.)

5.4.2 Bulk-Volume Meter

This procedure also involves submerging a core sample under mercury. The volume of mercury displaced by the sample further displaces a second liquid into an inclined, graduated glass tube. The bulk volume of the core is read directly off the graduated glass tube. (API RP 40, 1960, Sec. 3.312, p. 15; Sec. 3.57, p. 26.)

5.4.3 Boyle's Law Single-Cell Method Using the Kobe Porosimeter

The Kobe Porosimeter consists of a mercury pump, pressure gauge, gas inlet and outlet valves, and a sample chamber. Determination of the bulk volume of the sample requires filling the sample chamber containing the core sample with mercury. This leaves the core sample immersed in mercury. Bulk

volume is determined by subtracting the volume of mercury needed to fill the sample chamber without a sample from the volume of mercury needed to fill the chamber with a sample.

Grain volume is calculated by backing the mercury pump out to a "preset" volume. During this portion of the test the sample is not immersed in mercury. The pressure in the cell is vented to atmospheric pressure (P_1). Mercury is pumped into the sample cell (gas outlet valve shut) and the new pressure (P_2) is recorded. Knowing the volumes of mercury in the cell, the empty preset volume of the cell, and the pressures, Boyle's Law can be used to calculate grain volume ($P_1 \times V_1 = P_2 \times V_2$). Porosity is calculated from the bulk volume and the grain volume. Grain density is calculated from sample weight and grain volume. (API RP 40, 1960, Sec. 3.32211, p. 17; Sec. 3.5.10, p. 28.)

5.4.4 Washburn-Bunting Method

The Washburn-Bunting porosimeter measures the pore volume of the sample. The apparatus consists of a sample chamber with a graduated capillary tube on top with a stopcock for opening and closing the system to the atmosphere. A tube is attached to the bottom of the sample chamber which connects to a glass bulb filled with mercury. A sample is placed in the sample chamber. The procedure is begun by raising the mercury bulb to force mercury into the sample chamber, thereby immersing the sample, and up into the calibrated capillary. When the mercury is above the stopcock, the stopcock is closed. The mercury bulb is now lowered until the core sample is floating on the mercury inside the sample chamber. The sample is now under vacuum, and air from the pores of the sample fill the sample chamber and calibrated capillary. After a few minutes, atmospheric pressure is restored to the escaped air by raising the mercury bulb until the mercury level is equal to that in the capillary. The air volume in the calibrated capillary is equal to the pore volume of the rock sample. Improved accuracy is obtained by subtracting the air volume measured by using a solid billet in place of the core sample to account for adsorbed air on the sample surface. (API RP 40, 1960, Sec. 3.3221, p. 17; Sec. 3.5.12, p. 30.)

5.4.5 Dry Method for Grain Density and Pore Volume

In this procedure, the rock sample is extracted, dried, crushed, and sieved. A portion of the crushed rock that passes a 60-mesh screen and is caught by a 100-mesh screen is weighed and added into the sample cup of the apparatus. A mercury pump is then used to pump mercury into the sample cup. When the system pressure reaches the preset calibration value, the volume of injected mercury is recorded. By calculating the volume of the empty cup, the bulk volume of the sample in the cup can be determined. Using the sample weight, grain density can be computed. Using a companion rock sample, grain volume is computed by dividing the companion sample dry weight by the calculated grain density from the crushed sample. Pore volume can be determined by subtracting the grain volume from measured bulk volume of the sample. (API RP 40, 1960, Sec. 3.3211, p. 16; Sec. 3.58, p. 26.)

5.4.6 Wet Method for Grain Density and Pore Volume

The bulk volume is measured and the dry sample is weighed and crushed. The crushed sample is added to a calibrated volumetric flask containing a known volume of a suitable wetting fluid (e.g., toluene, water, etc.). The volume increase is equal to the grain volume of the crushed sample. The grain density can be calculated by dividing the weight of the crushed sample by its measured volume. The pore volume can be calculated by subtracting grain volume from bulk volume. (API RP 40, 1960, Sec. 3.3212, p. 16; Sec. 3.59, p. 27.)

5.4.7 Full Diameter Summation of Fluids Utilizing the Vacuum-Retort Method

This method was used extensively in "hard rock" regions (e.g. West Texas) where production is predominately from carbonate formations. Each full diameter test sample was weighed and then the gas filled pore space was pressure saturated with water. The sample was again weighed, with the weight increase equal to the gas volume. Fluids within the pore space were distilled from the core under partial vacuum, at a maximum temperature of 450°F (232°C). Fluids were collected in glassware immersed in an alcohol/dry ice bath maintained at –75°F (–59°C). This was to condense vapors and prevent loss through the vacuum system. Condensed volumes were read, and an oil correction factor was employed. The sample pore water was computed by subtracting the gas volume from total water collected in the condensing tube. Pore volume was computed by summing the gas, corrected oil, and pore water volumes. A bulk volume determined on the test sample was used for calculation of porosity (API RP 40, 1960, Sec. 4.21, p. 39; and 4.52, p. 42).

One of the prime disadvantages of this method was the condition of the cores after the distillation process. It was not uncommon for the cores to be black from coked oil and no cleaning process could restore the samples to a condition whereby they could be deemed suitable for use in further testing procedures (e.g., a Boyle's Law Porosity, Capillary Pressure Tests, Relative Permeability). For this and other reasons, the Subcommittee charged with the rewriting of Recommended Practice 40 elected not to record this as a recommended procedure.

5.5 ORGANIC-RICH ROCKS

In recent years, there has been considerable interest in the development of coalbed methane and gas shale reservoirs. Coals and gas shales are typically highly organic fractured

formations in which much of the gas-in-place is adsorbed to the organic material. Because adsorbed gas comprises a significant portion of the gas-in-place in these reservoirs, both the adsorbed gas content and how the gas is released (desorption/adsorption isotherms) are necessary for reserve estimates and production forecasts. Oil shales contain solid organic material, typically liquefied by application of heat to generate oil. Techniques to evaluate these type of deposits are discussed in 4.7. Porosity, as discussed herein, is not normally required.

5.5.1 Coal

There are no generally accepted methods in the industry for the laboratory measurements of porosity, permeability, or relative permeability of coal samples, nor are there published laboratory measurements that are accepted standards. Standard laboratory techniques for measurement of porosity in oil and gas bearing reservoir rock must, therefore, be modified when applied to coal used in coalbed methane studies. Following are comments and references reflecting current practices.

In production of methane from coal seams, "porosity" may be defined in terms of three distinct parameters. For calculation of coalbed methane gas-in-place, these are (a) the "in-situ gas content" of the coal, which is equivalent to the gas volume contained in the gas-filled porosity in a conventional gas reservoir. In addition, coal is a dual porosity system consisting of (b) matrix (micropore) porosity wherein the methane is adsorbed on the coal surface, and (c) cleat porosity (naturally occurring microfracture porosity). The cleat network provides the path and permeability for fluid flow.

5.5.1.1 In Situ Gas Content

The only method by which total in situ gas content of coalbeds can be measured directly is with the use of pressure core technology.[7] The volume of gas evolved from the coal core in a pressure core barrel is measured as a function of time, temperature, and pressure.

Gas content and desorption rate are often determined by canister desorption methods.[8] Coal samples utilized include cores obtained using conventional coring, drilled sidewall coring, wireline retrieval, and coal drill cuttings. The coal cores (or cuttings) are sealed in the canister and the volume of gas evolved from the sample is measured as above.[7] Gas content determination by canister desorption requires an estimation of the volume of gas lost while retrieving the samples to the surface and before sealing the cores in the canister.[9,10]

Gas content from conventional and sidewall core samples by the canister desorption method is much less expensive than pressure coring, and is the most commonly used technique. Even pressure cores are often placed in desorption canisters to complete the desorption process. Gas content from different methods or samples can be compared after normalizing the coal to an ash-free basis.[7] Since the gas evolved is not always 100 percent methane,[11] the composition of the gas must be analyzed to determine methane content.

5.5.1.2 Matrix Porosity

The matrix porosity of coal is generally larger than the cleat porosity. However, since the methane is adsorbed in the matrix rather than existing as a free gas, conventional oil or gas field matrix porosity measurements are seldom performed for coalbed methane applications. If conventional pore volume is required, equilibrium moisture[12] and helium Boyle's Law porosity[13] techniques can be used to measure matrix porosity.

Note: Any gas that adsorbs will yield erroneous matrix porosity values with the Boyle's Law technique. Helium, which is not appreciably adsorbed, is often used.

5.5.1.3 Cleat Porosity

Although the bulk of coal porosity is the matrix porosity, the cleat network provides the path and permeability for fluid flow. The measurement of cleat porosity in coal is, therefore, important in determining pore volume compressibility, as well as the saturations necessary for gas-water relative permeability or capillary pressure measurements.

Cleat porosity is a function of net confining pressure. Cleat porosity should be measured with the coal core in a uniform confining pressure. Jacketing materials commonly used for unconsolidated cores[16] are often used on coal cores.

Measurements of coal porosity have been made on coal cores dried in vacuum ovens.[14] Studies on coal weathering indicate such drying alters the coal structure,[15] yielding erroneous results. A suitable procedure for cleat porosity measurement on non-dried core is referred to as the miscible drive technique. In this procedure, the fluid saturating the core is displaced from the core by a second fluid with different physical properties, but which is miscible with the saturating fluid.[16]

The miscible drive technique may employ tracer concentration in water. A tracer that is adsorbed will yield erroneous results.[17] Within experimental error, the immiscible displacement with water-vapor-saturated helium of water from a water-saturated coal core yields the same results as the miscible drive tracer technique.[17]

Note: Coal cores often have residual methane in the matrix, which can affect porosity measurements. Saturation of coal core with helium (saturated with water vapor), followed by evacuation, has been used to remove the residual methane.[17]

5.5.2 Gas Producing Shale

In gas shales, such as the Devonian, gas may be stored as free gas in the matrix or fractures, and also exist as adsorbed gas on the organic and clay surfaces. Adsorbed gas may account for as much as 80 percent of the gas-in-place, emphasizing the need for adsorbed gas isotherms. Volumes of adsorbed gas in shale are $^1/_5$ to $^1/_{50}$ of that for coal, and measurement apparatus and procedures must be tailored to supply

accurate data. As is currently the case for coal, no industry standard procedures currently exist for conducting shale measurements.

Reported results of adsorbed gas isotherm measurements on shale samples indicate that a wide disparity in test conditions currently exists between testing laboratories.[18] These disparities result in varying adsorption results, and emphasize the need for persons using the data to clearly understand what is being measured and what is reported.

Simulators utilized in reservoir modeling often utilize as input adsorbed methane content versus pressure data. Data are typically reported in units of scf/ton. Consequently, it is important to know if the isotherm measured and reported is for adsorbed gas alone, or if it represents a total sorption isotherm, which includes adsorbed gas as well as gas stored as free gas in the matrix and/or fractures.

The presence of water reduces gas volumes adsorbed. Adsorption tests on dry rock, therefore, will over estimate gas reserves, and tests on rock samples containing an equilibrium moisture condition are preferred.

Note: Techniques to establish appropriate equilibrium moisture conditions representative of the reservoir are not yet defined. Consequently, many of the adsorption isotherms measured have been completed on shale "as received," or on dry rock.

Although the technology for measurement is still evolving, certain important elements affecting measurement results and reported data have been identified.[18] Engineers utilizing these data should discuss with the measurement laboratory the following key elements. Procedures should be established that are most likely to yield data representative of the reservoir and that are required in the mathematical simulator utilized.

5.5.2.1 Key Elements Affecting Measurement

Factors affecting the measurement include:

a. Core recovery techniques (including coring fluid).
b. Sample preservation technique.
c. Sample size and shape (crushed? sieved to what size?).
d. Sample drying conditions:
   1. "As received."
   2. Dried (at what temperature and how long?).
   3. At equilibrium conditions (what temperature and how long?).
e. Standard temperature and pressure used for reporting measured gas volumes as scf/ton.
f. Metric ton or U.S. ton of "as received," dried or equilibrium weight rock used as base for scf/ton.
g. Total sorbed (including free porosity) gas or adsorbed gas only isotherm.
h. Cell calibration technique.
i. Temperature measurement and control of cell.
j. Sample density reported (bulk or grain; wet or dry).
k. Was equilibrium reached at each adsorption level, or was test step terminated at selected adsorption time?
l. Sample volume tested.
m. Sample evacuated prior to testing.
n. Gas used in adsorption measurement (usually methane).
o. Gas used in "free gas volume" porosity measurement (usually helium).

5.5.2.2 Apparatus

An apparatus designed to determine volumetric adsorption is illustrated in Figure 5-9.[19] It resembles a commonly used double-cell porosimeter.

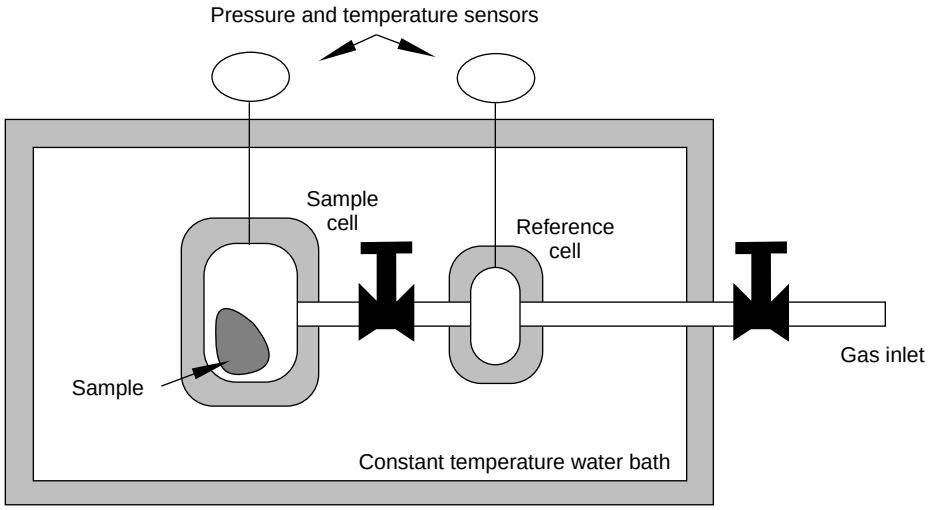


Figure 5-9—Volumetric Adsorption Measurement Method

5.5.2.2.1 Free (Fracture and Matrix) Porosity

Fracture and matrix void volume can be determined utilizing Boyle's Law double-cell porosimeter (see Figure 5-5) with helium as the gas phase.

5.5.2.2.2 Adsorption Isotherms

An isotherm measurement with helium gas is first completed. This yields free gas space with minimum adsorption. Methane gas is than charged into the reference cell and the gas is expanded into the sample chamber. The reference chamber is again isolated from the sample cell and is charged with gas at higher pressure. The procedure is repeated, and the gas adsorbed at the higher pressure level is calculated. This process is repeated until desired pressure levels are reached. The difference between the helium and methane isotherms represent adsorbed gas. Equations for calculation of isotherms, as well as an error analysis identifying factors that affect the accuracy of the measurement have been presented.[19]

5.5.3 Oil Shale

Evaluation of oil shales do not typically require measurement of rock porosity, as the oil recovered is generated from solid organic materials subjected to heat. See 4.7 for oil content determination.

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SECTION 6—PERMEABILITY DETERMINATION

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6 Permeability Determination

6.1 INTRODUCTION

Permeability is a property of a porous medium and is a measure of its ability to transmit fluids. The measurement of permeability of a porous rock, or stratum, is a measurement of the fluid conductivity of the particular material. Permeability is the fluid-flow analog of electrical or thermal conductivity. The reciprocal of permeability represents the viscous resistivity that the porous medium offers to fluid flow when low flow rates prevail. This condition of flow is customarily called "viscous flow," or formally, "Stokes flow". Measurement of the flow of fluid through a sample in a particular direction yields permeability of the sample in that direction. The permeability of a homogeneous, isotropic medium is the same everywhere, and in all directions. However, real rocks are neither perfectly homogeneous nor isotropic.

6.1.1 Definitions

6.1.1.1 Darcy Flow

The mid-nineteenth century experiments and studies of Darcy[1] led to an empirical expression of the relationships among the variables involved in the flow of fluids through porous media, known now as Darcy's Law. Briefly, Darcy's Law states that the volumetric rate of flow per unit cross-sectional area of permeable medium (the volumetric flux) is directly proportional to the potential gradient, and inversely proportional to the viscosity of the fluid. The coefficient of proportionality is permeability. Thus, the potential gradient required for Stokes flow is equal to the product of the fluid's viscosity and volumetric flux, divided by the permeability of the rock. The potential gradient for horizontal liquid flow, or in any direction for practical measurements with low density gas, is equal to the pressure gradient.

6.1.1.2 Forchheimer Inertial Resistance

The work of Forchheimer[2] at the beginning of the twentieth century showed that Darcy's Law is a limiting case, restricted to low volumetric fluxes. At higher fluxes, Forchheimer observed that the potential gradient required for a given volumetric flux is greater than that predicted by Darcy's Law by an amount proportional to the product of the fluid's density and the square of its volumetric flux. The coefficient of proportionality, β, is the inertial resistivity of the porous medium. Inertial energy dissipation is due to innumerable accelerations (i.e., time-rates of change in the direction and magnitude of velocity) that a fluid undergoes as it travels tortuous paths through a porous medium. These accelerations cause secondary flow patterns, in which, part of the flow energy is converted to heat through viscous shear.

In a petroleum reservoir, especially away from a producing or injection well, volumetric fluxes are generally so low that Darcy's Law is applicable. However, high near-wellbore fluxes associated with high pressure gradients can cause non-negligible inertial effects known as "rate-sensitive skins." These are particularly prevalent near gas-producing wellbores, and in perforations, where low gas viscosity permits very high fluxes.

In laboratory measurements of high permeability samples using gas (where inertial effects are most often observed), low volumetric fluxes require low pressure gradients, which can be difficult to measure accurately. Higher fluxes will cause the calculation of permeability to be low if Darcy's Law is employed. A procedure to estimate the maximum allowable pressure gradient as a function of permeability, to avoid significant permeability errors due to inertial resistance, is presented in 6.2.1.3. An alternative is to make multiple flow rate measurements and employ Forchheimer's equation to obtain both permeability and inertial resistivity of each sample. As a practical matter, this is most quickly accomplished with the pressure-transient technique presented in 6.4.1.1.

6.1.1.3 Klinkenberg Gas Slippage

Even when inertial effects are properly accounted for, the permeability of a porous medium to gas is dependent upon the mean free path of the flowing gas, hence among other things, upon its absolute pressure. This is due to a phenomenon known as slip, a fact first pointed out to the oil industry by Klinkenberg.[3] Gas slippage had been overlooked or ignored by prior investigators of permeability despite theory and experimental data for slip in the flow of gases through small capillary tubes presented in scientific literature as early as 1875.[4] When gas slippage is ignored, permeability calculated from the Forchheimer equation, or from Darcy's Law (provided that inertial resistance is negligible), is higher than that obtained using a non-reactive liquid. When expressed as a percentage, this difference is small for high permeability samples, but becomes progressively larger with decreasing permeability. It is minimized by using high mean pore pressures in gas permeability measurements.

To avoid the problem of obtaining pore-pressure-dependent gas permeabilities, Klinkenberg presented a method in which gas permeability measurements made at several different mean pore pressures can be extrapolated to infinite pore pressure. He showed that this extrapolated gas permeability (now called "Klinkenberg permeability", k_∞) is equal to the permeability obtained using a non-reactive liquid, such as a clean, refined hydrocarbon.

Permeability of rock samples, especially those that contain certain clays, can be altered when they are cleaned and dried in preparation for gas permeability measurements. Often, k_∞ is higher in these samples than permeability measured using aqueous solutions. Special cleaning and/or drying techniques may be required.

6.1.1.4 Liquids

The use of liquids for permeability measurements eliminates the problem of gas slippage, and at reasonable and usual flow rates, inertial resistance is generally negligible. Thus, Darcy's Law can be used directly to calculate permeability from a single flow rate measurement. However, potential permeability alteration from interaction of rock constituents and liquids (especially aqueous solutions), fines movement, and microbial plugging requires special attention. Also, the liquid remaining in a sample may have to be removed before other measurements can be performed. Because of these problems, most routine permeability measurements have been made using gases. However, for some samples, such as those sensitive to drying techniques, liquid permeability measurements are considered to be the only acceptable alternative.

6.1.1.5 Confining Stress Dependence

Permeability of a porous medium is sensitive to the magnitude of net compressive stresses to which the medium is subjected, and to its stress history. As confining stresses are increased in a sample holder, the rock's permeability decreases. The decrease observed, when confining stresses are increased from 200 or 300 psi to several thousand psi, varies considerably—from just a few percent for reasonably permeable, well-cemented samples, to an order of magnitude or more for low permeability samples that contain micro fractures. In general, it is desirable to approximate reservoir stresses in laboratory permeability measurements to obtain values closer to expected in situ reservoir permeabilities. Additional, smaller permeability decreases may also be observed as a function of time after confining stresses are raised—due to creep. Creep is most prevalent at compressive stresses just lower than those that cause sample failure.

6.1.2 Unit of Permeability

6.1.2.1 Traditional Unit

In order to make practical use of the concept that porous media possess the measurable property of permeability, an arbitrary standard unit, the "darcy," has been defined. A porous medium has a permeability of one darcy when a single-phase fluid of one centipoise viscosity that completely fills the voids of the medium will flow through it under "conditions of Stokes flow" at a rate of 1 cm^3/s per square centimeter of cross-sectional area under a pressure or equivalent hydraulic gradient of 1 atm/cm. "Condition of Stokes flow" shall mean simply that "the rate of flow be sufficiently low to be directly proportional to the pressure or hydraulic gradient." The unit of permeability (the darcy), as a coefficient of proportionality between physical quantities, possesses dimensions of length squared. Permeability represents a property of the medium alone and is independent of the fluid, except as hereinafter qualified. For convenience the millidarcy sub-unit (equal to 0.001 darcy) may be used. The spelling of the plural forms of the unit have been standardized and established in the literature as "darcys" and "millidarcys." Although other sub-units are not recommended, the microdarcy (equal to 0.001 millidarcy or 10^{-6} darcy) is often used in conjunction with low-permeability gas sands. Also, the nanodarcy (equal to 0.001 microdarcy or 10^{-9} darcy) is often referred to for very tight rocks, such as micro fractured granite.

6.1.2.2 SI Unit

The fundamental SI unit of permeability, m^2, is defined as follows: a permeability of one meter squared will permit a flow of 1 m^3/s of fluid of 1 Pa·s viscosity through an area of 1 m^2 under a pressure gradient of 1 Pa/m. One darcy equals 0.986923×10^{-12} m^2. Because of the small exponent, this is an awkward unit for common usage. The Society of Petroleum Engineers of AIME preferred permeability unit is the micrometer squared (μm^2). One darcy (the traditional unit) equals 0.986923 μm^2.

6.1.2.3 Typical Units Used in Hydrology

Most environmental engineers have a background in hydrology, in which the terms intrinsic permeability, hydraulic conductivity, and transmissivity are used. The physics of fluid flow that environmental engineers, core analysts, or petroleum engineers deal with is the same, but the language used by one group is often foreign to another group. This arises from different applications. Hydrologists most often deal with the flow of ground water in aquifers, in which water viscosity, density, and compressibility exhibit rather small variations. In these aquifers, it is often convenient to determine flow potential differences by measuring differences in liquid heads—the elevations to which water rises in small non-flowing test wells drilled into the aquifer. In the laboratory, an analogous measurement technique would be to attach a series of water manometers to a core sample through which water is flowing. The height of water in the manometer nearest the upstream end of the core would be greatest, and water-level elevations (from a horizontal reference) would be progressively lower further downstream.

In petroleum reservoirs, fluid properties vary widely, more than one fluid phase is usually present, and pressures are often too great to make manometric head measurements. Pressures are measured directly with pressure gauges or transducers. Thus, equations for these applications deal with pressures, not heads. Flow potentials must be calculated from

pressures, flow rates, and fluid and rock properties through appropriate governing partial differential equations. Solution often requires numerical finite difference approximations of these equations.

6.1.2.3.1 Flow Potential Differences vs. Pressure Differences

It is important to understand that flow potential difference, not pressure difference, is the driving force for fluid flow, to know what flow potential is, and to ascertain when pressure differences can legitimately be used in flow equations. Relationships between flow potential and pressure are illustrated by Figure 6-1. Two tanks, each open to atmospheric pressure at its top and fitted with manometer tubes at several depths, are connected together by a small pipe. Each is partially filled with water to the same depth. Pressure in each tank at the air-water interface is atmospheric, and increases with depth.

We observe that the water level in every manometer tube is the same, and except for a very small capillary rise in each tube, it is the same as the water level inside the tanks. This water level in the manometer tubes is a measure of the flow potential inside the tanks, that is the same at every depth, even though pressure increases with depth. If pressure, alone, were the driving force for flow, higher pressure water from the bottom of a tank would flow upward, toward lower pressure. This is not observed. If we assume that the density of water everywhere in each tank is constant (a very close approximation), then flow potential, φ, everywhere is:

$$\varphi = p - \rho g z / C_4 \quad (1)$$

Where:

p = pressure at depth z (below the water surface; z increases downward).
ρ = density of the water.
g = local acceleration of gravity.
C_4 = conversion factor (found in Table 6-1) to make units consistent.

However, pressure at any depth in the tank is:

$$p = p_1 + \rho g z / C_4 \quad (2)$$

Where p_1 is the pressure at the air-water interface, atmospheric pressure in this case. Thus, combining Equations 1 and 2, we see that $\varphi = p_1$, and flow potential is the same at any depth. It is also the same in both tanks; so no flow occurs within or between tanks.

Condition 1:
a. Both tanks are filled with water to same depth, and are open to atmospheric pressure.
b. Water pressure increases in both tanks with increasing depth.
c. However, flow potential (indicated by water levels in manometer tubes) is the same for all depths, and is the same in both tanks.
d. Therefore, no flow occurs within or between tanks.

Condition 2:
a. Air pressure above water in left tank is increased. Then vent is capped.
b. Flow potential in left tank is increased as indicated by increased water levels in manometer tubes there.
c. Water flows from left tank to right tank until water levels in manometer tubes are the same for both tanks.

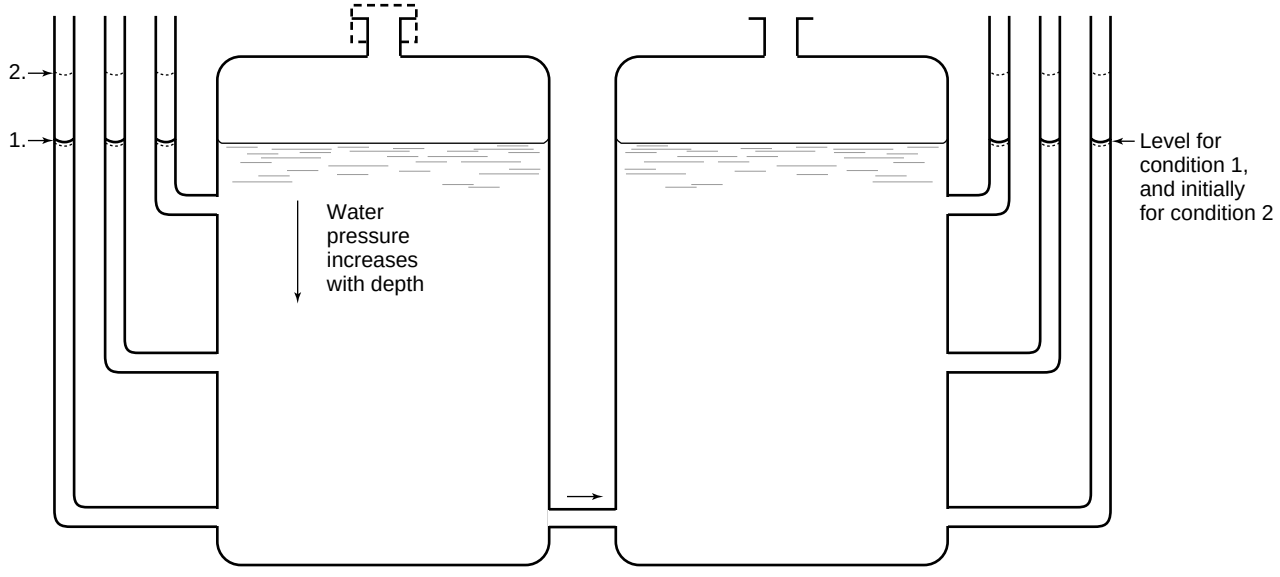


Figure 6-1—Illustration of Flow Potential vs. Pressure Differences

Table 6-1—Constants in Forchheimer or Darcy Equation with Various Sets of Units

Variable or Constant	Units or Values of Constants			
	SI	Preferred SPE	Traditional	Common Usage
A, area perpendicular to flow	m^2	m^2	cm^2	cm^2
b, Klinkenberg slip factor	Pa	Pa	atm	psi
D, diameter of sample	m	m	cm	cm
g, gravitational acceleration	m/s^2	m/s^2	cm/s^2	cm/s^2
k, permeability	m^2		darcy	millidarcy
L, length of sample	m	m	cm	cm
M, gas molecular weight	kg/kg-mole	kg/kg-mole	g/g-mole	g/g-mole
P, absolute pressure	Pa	Pa	atm	psia
p, gauge pressure	Pa	Pa	atm	psig
Δp, differential pressure	Pa	Pa	atm	psi
q, volumetric flow rate				
r, radius of sample or seal	m	m	cm	cm
S, compressive storage				
s, flow-direction distance	m	m	cm	cm
T, absolute temperature	K	K	K	K
V_p, pore volume				
V_b, total reservoir volume				
v_s, volumetric flux	m/s	m/s	cm/s	cm/s
β, inertial resistivity	m^{-1}	m^{-1}	cm^{-1}	$ft.^{-1}$
ρ, gas or liquid density	kg/m^3	kg/m^3	g/cm^3	g/cm^3
μ, gas or liquid viscosity	Pa·s	Pa·s	cp	cp
R, gas-law constant	8314	8314	82.05	1205.8
C_1, in Darcy or Forch. eq.	1.0	1.0	1.0	6.8046E-2
C_2, in Darcy or Forch. eq.	1.0	1.0E+12	1.0	1000.
C_3, constant in Forch. eq.	1.0	1.0	9.8692E-7	3.2379E-8
C_4, constant in eqs. with g	1.0	1.0	1.0133E+6	68 950.

Now, suppose air is pumped into the left tank through its vent so that air pressure above the water is greater than atmospheric pressure. Then the vent is capped. We would observe that water levels in all manometers attached to the left tank have risen equally. Except for the amount of water required to increase levels in the tubes, the water level inside the tank is unchanged. Initially, conditions in the right tank are unchanged. From the higher manometer levels in the left tank (indicated by dashed lines in Figure 6-1), relative to those in the right tank, we conclude that flow potential is higher in the former, even though water levels inside the two tanks are the same. Indeed, water flows from left to right until manometer levels are the same for both tanks. When flow ceases, the water level inside the left tank will be lower than in the right tank.

In laboratory single-phase gas permeability measurements, "gas head" differences are generally smaller than the accuracy of pressure measurements, and can be ignored. Therefore pressure differences or pressure gradients are very close approximations of flow potential differences or gradients in any flow orientation (horizontal or vertical). Conditions for single-phase liquid measurements are discussed in detail in 6.2.2. Pressure differences or gradients can be substituted for flow potential differences or gradients only in special, restricted situations.

6.1.2.3.2 Intrinsic Permeability

Intrinsic Permeability, k, as used by hydrologists is identical to permeability defined in 6.1.2.2, even though it is defined in terms of heads and kinematic viscosity, v, rather than dynamic viscosity, μ, used in the Darcy equation as defined in 6.1.2.1. Kinematic viscosity is equal to dynamic viscosity divided by density of the fluid. A porous medium has an intrinsic permeability of one unit of length squared if it will transmit in unit time a unit volume of fluid of unit kinematic viscosity through a cross section of unit area measured at right angles to the flow direction under a unit potential gradient. If SI units are used, intrinsic permeability has the unit of m^2.

6.1.2.3.3 Hydraulic Conductivity

Hydraulic conductivity, K, is not solely a property of a porous medium. It depends also upon the kinematic viscosity of the flowing fluid, and is appropriate only for ground water aquifers. Its units are those of velocity, usually feet per day or meters per day. A medium has a hydraulic conductivity of

unit length per unit time if it will transmit in unit time a unit volume of ground water at the prevailing viscosity through a cross section of unit area, measured at right angles to the direction of flow, under a hydraulic gradient of unit change in head through unit length of flow. Hydraulic conductivity is related to permeability (or intrinsic permeability) by:

$$K = \frac{k\rho g}{\mu} \quad (3)$$

Where k is permeability, and the other symbols are defined above. Any consistent set of units may be used in Equation 3. With SI units, K will have the unit of meters per second.

6.1.2.3.4 Transmissivity

The transmissivity, T, of a ground water aquifer includes the thickness of the aquifer, b, and the kinematic viscosity of the confined water. Transmissivity is the rate at which water of the prevailing kinematic viscosity is transmitted through a unit width of the aquifer under a unit hydraulic gradient. It has the unit of length squared per unit time, often ft^2/day, or m^2/day. Transmissivity is equal to Kb. It can be calculated from permeability by:

$$T = \frac{k\rho g b}{\mu} \quad (4)$$

Definitions for intrinsic permeability, hydraulic conductivity, and transmissivity are taken from Lohman.[42] Conversion factors for several sets of units can be found in *Ground Water* (1993).[43]

6.1.3 Terminology

6.1.3.1 Specific Permeability

The definition of the standard unit of permeability requires that the porous medium contain only one homogeneous, single-phase fluid. The property of the medium so determined is properly termed specific permeability; but it shall simply be called permeability, and denoted by the symbol k. By this definition, the term permeability becomes a property solely of the porous medium; and the numerical value of permeability is constant and independent of the fluid used in the measurement. Whenever certain minerals and fluids are brought in contact and interaction results, or material is deposited from the fluid, this phenomenon, in effect, produces a new medium. The permeability of the new medium may differ from that of the unaltered medium. Whenever this occurs, it should be indicated clearly by appropriate references to both medium and fluid, e.g., "the permeability of Woodbine sand to salt water (fresh water, crude oil, heptane, etc., as the case may be)."

Uncorrected gas permeabilities are precluded from the above definition due to gas slippage effects. Gas permeabilities, which shall be called k_g (or k_{air}, k_{N2}, k_{He}, etc.) depend upon the particular gas used in the permeability determination, and upon its mean pore pressure. Both must be specified to define the permeability of the medium. Only the Klinkenberg, slip-corrected gas permeability, when unaffected by high velocity (Forchheimer) effects, is constant and independent of the fluid used in the measurement.

Because the stress condition under which a measurement is made can have a significant effect upon permeability, it also should be specified. Examples are: k_{air} with a mean pore pressure of 32.3 psia and a radial confining stress of 400 psig; or k_∞ at a net hydrostatic stress of 5,000 psi. The first specification implies that a gas permeability was obtained using air at an average absolute pore pressure of 32.3 psia, and that a pressure of 400 psig was applied to the rubber boot of the sample holder. The net radial stress is therefore 400 + 14.7 – 32.3, or about 382 psi. The net axial stress is unknown. In the second example, a Klinkenberg (slip-corrected) permeability was obtained that is independent of the gas used or its mean pore pressure (except as pore pressure affects net stress). Equal magnitude radial and axial ("hydrostatic") net stresses of 5,000 psi were applied during the measurement.

6.1.3.2 Effective and Relative Permeability

This manual of routine recommended practices deals only with single-phase permeability measurements. Therefore, effective permeability and relative permeability will only be defined. The effective permeability of a porous medium is a measure of its fluid conductivity to a particular phase of a multi-phase fluid system residing within the medium, where the saturation of each phase (the fraction of its total effective pore volume that is filled by each phase) is specified. Relative permeability is the ratio of the effective permeability of a particular fluid phase, at definite saturations of all phases, to some arbitrary reference permeability. The reference permeability can be the sample's specific permeability, its Klinkenberg permeability, the effective permeability of one of the fluid phases at a specified saturation condition, etc. The particular reference permeability used must be specified.

Book three of the five volume, *History of Petroleum Engineering* (API, 1961),[39] is a rich reference source for early work on laboratory permeability measurements, including the pioneering efforts of Fancher, Lewis, and Barnes,[40] and of Wyckoff, Botset, Muskat, and Reed.[41]

6.2 THEORY

Single-phase permeability measurements can be separated into four major categories: those utilizing flowing gas or liquid under steady-state or unsteady-state (transient) conditions. Equations for steady-state flow of gases and liquids are presented in this section. Several practical variations, each with particular advantages and limitations, will be outlined in 6.3, and the theory and application of transient pressure gas

and liquid measurements are presented in 6.4. These are all considered to be direct measurements.

The permeability of a porous medium can also be inferred from empirical correlations using indirect measurements, such as those of inter granular porosity and grain size (Kozeny[5] and Carman[6]); porosity and irreducible water saturation (Wyllie and Rose[7]); pore-throat and pore-size distribution by mercury injection or capillary pressure measurements (Swanson[8], Katz and Thompson[9], etc.); effective electrical conductivity (Archie[10], Ondracek[11]); transmissivity of acoustic waves (Biot[12]); electrokinetic emf (Moran[13]); etc. Because these can be less accurate and often more time consuming than making the direct measurements, they will not be considered here.

All measurements are assumed to be conducted under isothermal conditions, which may require considerable technique and effort to achieve under certain circumstances. Also, even though temperature can affect permeability, most routine permeability measurements are made at, or near, room temperature.

Tables 6-2 and 6-3 are quick selection and reference guides for gas and liquid permeability determinations, respectively, using both steady-state and transient techniques. They list major advantages and disadvantages of each technique, its approximate permeability range, and particular application. They are intended to be a starting point only, and the reader is cautioned to be aware of details given in sections referenced by these tables.

Table 6-2—Quick Selection and Reference Guide for Permeability Measurements Using Gases*

Reference Section Numbers	Type of Measurement	Approx. Perm. Range, md	Apparatus or Application	Major Advantages	Major Limitations
6.3.1.1 6.3.1.1.1.1	Axial flow, steady state in core plugs	0.1-10,000	Low pressure apparatus with manometers, orifice flow meters	Low capital cost; simple manual system; workhorse for decades; large data base for comparison	Labor intensive; high operating cost; low-stress perms; no slip correction; must check for inertial resistance
6.3.1.1 6.3.1.1.1.2	Axial flow, steady state in core plugs	0.1-10,000	Apparatus with electronic sensors, high pressure. core holder	Can be automated; reservoir stresses can be approximated; better precision and accuracy than with manual system	Must make multiple measurements for gas slippage correction; must check to ensure negligible inertial resistance
6.4.1.1 B.6.8.2	Axial flow, pressure falloff in core plugs	0.001-30,000	Wide range; med. to high stress measurements with corrections for b and β	Well adapted for automation; no flow meters required; can yield reservoir-condition perms (k_∞), and k_g	Higher capital cost for automated system with high accuracy pressure transducers and data acquisition system
6.4.1.3 D.6.8.4	Axial flow, pulse-decay in core plugs	.00001-0.1	High stress apparatus for very low perms.	Only method for ultra-low perms; well adapted for automation; porosity can be determined in same apparatus	Requires high pressure, leak-tight system with high quality transducers and data acquisition system—higher capital cost
6.3.1.2	Probe perm., s.s., on whole core	1-10,000	Zero stress, high density, localized measurements for heterogeneous cores	No plug preparation required (core slabbing recommended); relatively fast; can be automated or made portable	Zero stress, non slip corrected perms are high at low end of range; prone to high inertial resistance at high end
6.4.1.2 C.6.8.3	Probe perm., pressure falloff on whole core	0.001-30,000	Zero stress, high density, localized measurements for heterogeneous cores	No plug preparation required (core slabbing recommended); very fast; automated; corrected for b, β	Zero stress perms are high, especially at low end of range; higher capital cost for automated system
6.3.1.3	Transverse, s.s. perm. in whole core	0.02-500	Directional perm. in whole core (or plug) for k_{max} and k_{90*}	Can measure "horizontal" perm. in various directions; averaging obtained using whole-core sample.	Cleaning and preparation of whole core sample more expensive; only k_g obtained without multiple measurements
6.3.1.4	Radial, s. s. perm. in whole core	0.01-250	Average permeability in all radial directions in whole core samples	Measures average "horizontal" permeability in large sample	Difficult to prepare samples; no radial stress; perm. critically dependent on condition of central "wellbore"

*Major advantages of using gas rather than liquid:

a. Easy to use—does not require special saturation techniques.
b. Non-reactive with rock; non-corrosive to equipment.
c. No post-measurement cleanup required.
d. Less prone than liquid to mobilizing fines in rock sample.
e. Does not support microbial growth, nor require special filtration.

Major disadvantages:

a. Requires correction for gas slippage—especially with lower perms.
b. Prone to significant high-velocity inertial resistance in high perm. rock.
c. Necessary leak-tightness harder to achieve than with liquids.
d. In some cases, may be less representative of permeability in reservoir.

Table 6-3—Quick Selection and Reference Guide for Permeability Measurements Using Liquids

Reference Section Numbers	Type of Measurement	Approx. Perm. Range, md	Apparatus or Application	Major Advantages	Major Limitations
6.3.2.2	Axial, s.s. flow in plugs using liquid heads	1000-40,000	Gravity flow of liquid through high perm., cylindrical core plugs	Simple, low cost equipment (but does require electronic balance for flow rate determination)	Low back pressure—difficult to ensure that no gas remains in core plug.
6.3.2.1	Axial flow, steady state in core plugs	0.1-20,000	Apparatus with electronic sensors, high pressure. core holder, imposed	Can be automated; reservoir stresses can be approximated; may be most representative measurement of perm. in reservior	Corrosion resistant, moderate to high pressure pumping and control equipment is expensive
6.4.2.1 E.6.8.5	Axial flow, pulse-decay in core plugs	.00001-0.1	High stress apparatus for very low perms.	Only method for ultra-low perms; well adapted for automation; no flow meters required: rate is calc. from Δp & t	Requires high pressure, leak-tight system with high quality transducers and data acquisition system—high capital cost
6.3.2.1 6.3.1.3.1	Transverse, s.s. flow in whole cores	0.005-500	Directional perm. in whole core (or plug) for k_{max} and $k_{90°}$	Can measure "horizontal" perm. in various directions; averaging obtained using whole-core sample.	Same as for s.s. axial flow (above); cleaning, preparation and handling of whole core sample more expensive
6.3.1.4 6.3.1.4.2	Radial, steady state flow in whole cores	0.002-250	Average permeability in all radial directions in whole core samples	Measures average "horizontal" permeability in large sample	Same as above; difficult to prepare samples; no radial stress; perm. critically dependent on condition of central "wellbore"

6.2.1 Steady-State Permeability Equations For Gases

The general equation for steady-state gas permeability measurements will be presented first. It includes provision for gas slippage and inertial resistance effects, which become somewhat complicated and require more measurements than is usually practicable using steady-state measurements. However it serves to unify the theory of gas measurements and provides principles and guidelines for use of the Darcy equation, which is the basis for the practical steady-state cases that follow.

Unlike liquids, gases are highly compressible. Also, under normal laboratory conditions, the density of gas is sufficiently low that gravity effects may be ignored in making permeability measurements. The viscosity of air, nitrogen, or helium (see Tables 6-4, 6-5, and 6-6, respectively) is less than $^1/_{50}$ that of water at room temperature. Therefore, volumetric fluxes of gases are correspondingly higher for a given pressure gradient, and can deviate from conditions of Stokes flow, demanded by Darcy's Law. Finally, the phenomenon of gas slippage, or the Klinkenberg effect, requires special treatment. Because of these differences, the point of departure for developing equations pertaining to the measurement of permeability using gases is the differential form of the Forchheimer equation, into which the Klinkenberg relationship will be inserted:

$$\frac{-dP}{ds} = \frac{C_2 \mu q_s}{C_1 A k_g} + \frac{C_3 \beta \rho_s^{\,2}}{C_1 A^2} \qquad (5)$$

Where:

s = distance along the direction of flow.
q_s = volumetric rate of gas flow through area A per unit time.
A = area perpendicular to the flow of gas.
$\frac{dP}{ds}$ = pressure gradient along s to which q_s / A refers.
μ = dynamic gas viscosity.
k_g = apparent permeability of the medium to a particular gas.
β = coefficient of inertial resistivity.
ρ = gas density.

The constants C_1, C_2, and C_3 allow for various sets of dimensions to be used in the gas-flow equations that follow. Table 6-1 shows values of these constants for SI, preferred-SPE, traditional, or common-usage dimensions. Note that an upper case P appears in the equation above. This non-standard symbol denotes that the pressure is *absolute*. A lower-case p is used to denote a *gauge* or *differential* pressure in any equation below. These conventions have been adopted in this document because of the confusion between the two pressures, and the frequent error made when a measured gauge-pressure is not converted to an absolute pressure when required. Thus, in keeping with this convention:

$$P = p + P_a \qquad (6)$$

Where P_a is the true ambient barometric pressure, converted to the proper units (not the barometric pressure from the local airport, which has been converted to sea-level pressure).

The density of a real gas is:

$$\rho = \frac{MP}{zRT} \qquad (7)$$

Table 6-4—Viscosity of Air (for Degrees F and C) at One Atmosphere*

Viscosity of air at one atmosphere, micropoises												
	Temperature, °F						Temperature, °C					
plus	50°F	60°F	70°F	80°F	90°F	100°F	0°C	10°C	20°C	30°C	40°C	50°C
0	176.8	179.6	182.3	185.0	187.7	190.4	171.8	176.8	181.8	186.6	191.4	196.2
1	177.1	179.9	182.6	185.3	188.0	190.6	172.3	177.3	182.3	187.1	191.9	196.6
2	177.4	180.1	182.9	185.6	188.2	190.9	172.8	177.8	182.8	187.6	192.4	197.1
3	177.7	180.4	183.1	185.8	188.5	191.2	173.3	178.3	183.2	188.1	192.9	197.6
4	177.9	180.7	183.4	186.1	188.8	191.4	173.8	178.8	183.7	188.6	193.3	198.0
5	178.2	181.0	183.7	186.4	189.0	191.7	174.3	179.3	184.2	189.0	193.8	198.5
6	178.5	181.2	184.0	186.6	189.3	192.0	174.8	179.8	184.7	189.5	194.3	198.9
7	178.8	181.5	184.2	186.9	189.6	192.2	175.3	180.3	185.2	190.0	194.7	199.4
8	179.0	181.8	184.5	187.2	189.8	192.5	175.8	180.8	185.7	190.5	195.2	199.9
9	179.3	182.1	184.8	187.4	190.1	192.8	176.3	181.3	186.2	191.0	195.7	200.3

*Calculated from Sutherland's equation:

$$\mu_{air} = \frac{14.969T^{1.5}}{T + 120}$$

Where: T is absolute temperature, degrees Kelvin (degrees Celsius + 273), and μ_{air} is the viscosity of air at one atmosphere pressure, micropoises. Multiply by 1.0 E-04 to convert to centipoises, or by 1.0 E-07 to convert to Pa·s. Constants are based on work of Montgomery[35] and Birge[36].

Table 6-5—Viscosity of Nitrogen (for Degrees F and C) at One Atmosphere*

Viscosity of air at one atmosphere, micropoises												
	Temperature, °F						Temperature, °C					
plus	50°F	60°F	70°F	80°F	90°F	100°F	0°C	10°C	20°C	30°C	40°C	50°C
0	171.3	173.8	176.4	178.9	181.4	183.8	166.6	171.3	175.9	180.4	184.8	189.2
1	171.5	174.1	176.6	179.1	181.6	184.1	167.1	171.7	176.3	180.8	185.2	189.6
2	171.8	174.3	176.9	179.4	181.9	184.3	167.5	172.2	176.8	181.3	185.7	190.0
3	172.0	174.6	177.1	179.6	182.1	184.6	168.0	172.7	177.2	181.7	186.1	190.5
4	172.3	174.8	177.4	179.9	182.3	184.8	168.5	173.1	177.7	182.2	186.6	190.9
5	172.5	175.1	177.6	180.1	182.6	185.1	168.9	173.6	178.1	182.6	187.0	191.3
6	172.8	175.3	177.9	180.4	182.8	185.3	169.4	174.0	178.6	183.0	187.4	191.8
7	173.1	175.6	178.1	180.6	183.1	185.5	169.9	174.5	179.0	183.5	187.9	192.2
8	173.3	175.9	178.4	180.9	183.3	185.8	170.3	174.9	179.5	183.9	188.3	192.6
9	173.6	176.1	178.6	181.1	183.6	186.0	170.8	175.4	179.9	184.4	188.7	193.0

*Calculated from Sutherland's equation:

$$\mu_{N_2} = \frac{13.85T^{1.5}}{T + 102}$$

Where: *T* is absolute temperature, degrees Kelvin (degrees Celsius + 273), and μ_{N_2} is the viscosity of nitrogen at one atmosphere pressure, micropoises. Multiply by 1.0 E-04 to convert to centipoises, or by 1.0 E-07 to convert to Pa·s. Constants are based on the work of Licht and Stechert[37].
The viscosity of nitrogen at temperatures reasonably close to 25°C and pressures to 253 atmospheres can be calculated from:

$$\mu_{N_2}[T, P] = \mu_{N_2}[T, 1] - 0.12474 + 0.123688P + 1.05452\text{E-}03P^2 - 1.5052\text{E-}06P^3$$

Where: $\mu_{N_2}[T, 1]$ is the viscosity of nitrogen at one atmosphere pressure (from the tables above), micropoises, and *P* is pressure, atmospheres. The pressure dependence is fitted to the data of Gracki, *et al.*[34] to within a maximum deviation of 0.1%. Pressure dependence at 0°C and 50°C is nearly the same as at 25°C.

Table 6-6—Viscosity of Helium (for Degrees F and C) at One Atmosphere*

Viscosity of air at one atmosphere, micropoises												
	Temperature, °F						Temperature, °C					
plus	50°F	60°F	70°F	80°F	90°F	100°F	0°C	10°C	20°C	30°C	40°C	50°C
0	191.7	194.2	196.8	199.3	201.8	204.4	187.0	191.7	196.3	200.8	205.4	209.8
1	191.9	194.5	197.0	199.6	202.1	204.6	187.5	192.1	196.7	201.3	205.8	210.3
2	192.2	194.7	197.3	199.8	202.3	204.9	187.9	192.6	197.2	201.7	206.3	210.7
3	192.4	195.0	197.5	200.1	202.6	205.1	188.4	193.1	197.6	202.2	206.7	211.2
4	192.7	195.3	197.8	200.3	202.9	205.4	188.9	193.5	198.1	202.7	207.1	211.6
5	193.0	195.5	198.1	200.6	203.1	205.6	189.3	194.0	198.6	203.1	207.6	212.0
6	193.2	195.8	198.3	200.8	203.4	205.9	189.8	194.4	199.0	203.6	208.0	212.5
7	193.5	196.0	198.6	201.1	203.6	206.1	190.3	194.9	199.5	204.0	208.5	212.9
8	193.7	196.3	198.8	201.3	203.9	206.4	190.7	195.4	199.9	204.5	208.9	213.4
9	194.0	196.5	199.1	201.6	204.1	206.6	191.2	195.8	200.4	204.9	209.4	213.8

*Calculated from:

$$\mu_{He} = 187.0\left(\frac{T}{273.1}\right)^{0.685}$$

Where: T is absolute temperature, degrees Kelvin (degrees Celsius + 273), and μ_{He} is the viscosity of helium at one atmosphere pressure, micropoises. Multiply by 1.0 E-04 to convert to centipoises, or by 1.0 E-07 to convert to Pa·s. Constants are from Chapman and Cowling[38].
According to the data of Gracki, *et al.*[34] the viscosity of helium at 25°C passes through a very shallow minimum with pressure. At 37 atm. it is 0.43 percent lower than at 1 atm., and at 158 atm., its viscosity is 0.17 percent higher than at 1 atm.

Where *z* is the gas deviation factor, which is equal to 1.0 for an ideal gas. Values of *z* as functions of temperature and pressure are given in Tables 6-7, 6-8, and 6-9 for air, nitrogen, and helium, respectively. They are calculated from the Beattie-Bridgeman equation of state[14]. See Table 6-1 for values of the universal gas-law constant, *R*, for the various sets of units. *M* is the gas molecular weight. Temperature in Equation 7 is absolute, in degrees Kelvin:

$$\begin{aligned} T &= °\mathrm{C} + 273.15 \\ &= (°\mathrm{F} - 32)/1.8 + 273.15 \end{aligned} \quad (8)$$

6.2.1.1 Treatment of the Gas Slippage Factor

The Klinkenberg relationship, written as a point function (i.e., pertaining to a particular point in a sample, not an average value) is:

$$k_g = k_\infty\left(1 + \frac{b}{P}\right) \quad (9)$$

Note that k_g depends upon pressure. Therefore it cannot be treated as a constant when the Forchheimer (or Darcy) equation is integrated. In Equation 9, *b* must have the same units as those of the pressure used.

Unfortunately, the gas slippage factor, *b*, is partly a rock property and partly a gas property, which has caused confusion and some problems. For example, a *b* measured using air, but not indicated as such, might be misused in calculations involving a different gas with significantly different properties. It is a fairly simple procedure to separate the gas properties from the slip factor, so that only the rock-dependent property remains. According to Klinkenberg[3], *b* is related to the mean free path of gas molecules, $\bar{\lambda}$, by the relationship:

$$\bar{\lambda} = \frac{br}{4cP} \quad (10)$$

But, from the kinetic gas theory[15], the mean free path is given by:

$$\bar{\lambda} = \frac{1.881\mu}{P}\sqrt{\frac{RT}{M}} \quad (11)$$

In Equations 10 and 11, any consistent units may be used. From these two equations, and adjusting for the units given below, we obtain:

$$b = \left[99.5\mu\sqrt{\frac{T}{M}}\right]\left(\frac{c}{r}\right) \quad (12)$$

Where:

P = absolute gas pressure.
R = universal gas law constant.
b = Klinkenberg gas slippage factor, psi. (The constant 99.5 becomes 6.77 if b has the unit of atm).
μ = gas viscosity, cp.

Table 6-7—Gas Law Deviation Factors for Air, Calculated from Beattie-Bridgemann Equation of State[14]

P,	Z-Factors for Air at Temperatures (Degrees F) of:							
psia	55	60	65	70	75	80	85	90
0	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000
10	0.99968	0.99970	0.99972	0.99973	0.99975	0.99977	0.99979	0.99980
20	0.99935	0.99939	0.99943	0.99947	0.99951	0.99954	0.99957	0.99961
30	0.99904	0.99910	0.99915	0.99921	0.99926	0.99931	0.99936	0.99941
40	0.99872	0.99880	0.99888	0.99895	0.99902	0.99909	0.99916	0.99922
50	0.99840	0.99850	0.99860	0.99869	0.99878	0.99887	0.99895	0.99903
60	0.99809	0.99821	0.99833	0.99844	0.99854	0.99865	0.99875	0.99884
70	0.99778	0.99792	0.99806	0.99818	0.99831	0.99843	0.99854	0.99866
80	0.99747	0.99763	0.99779	0.99793	0.99808	0.99821	0.99834	0.99847
90	0.99717	0.99735	0.99752	0.99769	0.99784	0.99800	0.99815	0.99829
100	0.99687	0.99706	0.99726	0.99744	0.99762	0.99779	0.99795	0.99811
110	0.99657	0.99678	0.99699	0.99720	0.99739	0.99758	0.99776	0.99793
120	0.99627	0.99651	0.99673	0.99695	0.99717	0.99737	0.99757	0.99776
130	0.99597	0.99623	0.99648	0.99671	0.99694	0.99716	0.99738	0.99758
140	0.99568	0.99596	0.99622	0.99648	0.99672	0.99696	0.99719	0.99741
150	0.99539	0.99568	0.99597	0.99624	0.99651	0.99676	0.99701	0.99724
160	0.99510	0.99541	0.99572	0.99572	0.99601	0.99601	0.99629	0.99656
170	0.99481	0.99515	0.99547	0.99578	0.99608	0.99637	0.99664	0.99691
180	0.99453	0.99488	0.99522	0.99555	0.99587	0.99617	0.99646	0.99675
190	0.99425	0.99462	0.99498	0.99533	0.99566	0.99598	0.99629	0.99659
200	0.99397	0.99436	0.99474	0.99510	0.99545	0.99579	0.99611	0.99643
210	0.99369	0.99410	0.99450	0.99488	0.99525	0.99560	0.99594	0.99627
220	0.99342	0.99385	0.99426	0.99466	0.99505	0.99542	0.99577	0.99612
230	0.99315	0.99360	0.99403	0.99445	0.99485	0.99523	0.99560	0.99596
240	0.99288	0.99335	0.99380	0.99423	0.99465	0.99505	0.99544	0.99581
250	0.99261	0.99310	0.99357	0.99402	0.99445	0.99487	0.99528	0.99566
260	0.99235	0.99285	0.99334	0.99381	0.99426	0.99470	0.99511	0.99552
300	0.99132	0.99190	0.99246	0.99299	0.99351	0.99401	0.99449	0.99496
400	0.98891	0.98968	0.99041	0.99112	0.99180	0.99246	0.99.09	0.99370
500	0.98676	0.98770	0.98861	0.98948	0.99032	0.99113	0.99190	0.99265
600	0.98486	0.98598	0.98705	0.98808	0.98907	0.99002	0.99094	0.99183
700	0.98322	0.98450	0.98573	0.98692	0.98805	0.98915	0.99020	0.99122
800	0.98185	0.98329	0.98467	0.98600	0.98728	0.98850	0.98968	0.99082
900	0.98074	0.98233	0.98386	0.98532	0.98673	0.98809	0.98939	0.99065
1000	0.97989	0.98163	0.98329	0.98489	0.98642	0.98790	0.98932	0.99069
1100	0.97931	0.98118	0.98297	0.98470	0.98635	0.98794	0.98947	0.99094
1200	0.97900	0.98099	0.98290	0.98474	0.98651	0.98821	0.98984	0.99141
1300	0.97894	0.98105	0.98308	0.98503	0.98690	0.98869	0.99042	0.99208
1400	0.97915	0.98137	0.98350	0.98555	0.98751	0.98940	0.99122	0.99296
1500	0.97961	0.98193	0.98416	0.98630	0.98835	0.99033	0.99223	0.99405
1600	0.98032	0.98273	0.98505	0.98728	0.98941	0.99147	0.99344	0.99534
1700	0.98128	0.98378	0.98618	0.98848	0.99069	0.99281	0.99485	0.99682
1800	0.98248	0.98505	0.98752	0.98990	0.99218	0.99436	0.99647	0.99849
1900	0.98391	0.98656	0.98909	0.99153	0.99387	0.99611	0.98827	1.00034
2000	0.98558	0.98828	0.99088	0.99337	0.99576	0.99805	1.00026	1.00238
2100	0.98747	0.99022	0.99287	0.99540	0.99784	1.00018	1.00243	1.00460
2200	0.98957	0.99237	0.99506	0.99764	1.00011	1.00249	1.00478	1.00698
2300	0.99189	0.99472	0.99744	1.00006	1.00257	1.00498	1.00730	1.00953
2400	0.99440	0.99727	1.00002	1.00266	1.00520	1.00763	1.00998	1.01223
2500	0.99711	1.00000	1.00277	1.00543	1.00799	1.01045	1.01282	1.01509
2600	1.00000	1.00291	1.00570	1.00838	1.01095	1.01343	1.01581	1.01810
2700	1.00307	1.00599	1.00879	1.01148	1.01407	1.01655	1.01894	1.02124
2800	1.00631	1.00923	1.01204	1.01474	1.01733	1.01983	1.02222	1.02794
2900	1.00971	1.01264	1.01545	1.01815	1.02074	1.02323	1.02563	1.02794
3000	1.01327	1.01619	1.01900	1.02169	1.02428	1.02678	1.02917	1.03147

P,	Z-Factors for Air at Temperatures (Degrees C) of:							
psia	0	5	10	15	20	25	30	35
0	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000
10	0.99957	0.99962	0.99966	0.99969	0.99973	0.99976	0.99979	0.99982
20	0.99915	0.99923	0.99931	0.99939	0.99946	0.99952	0.99958	0.99964
30	0.99873	0.99886	0.99897	0.99908	0.99919	0.99928	0.99937	0.99946
40	0.99831	0.99848	0.99864	0.99878	0.99892	0.99905	0.99917	0.99928
50	0.99790	0.99811	0.99830	0.99848	0.99866	0.99882	0.99897	0.99911
60	0.99748	0.99773	0.99797	0.99819	0.99839	0.99859	0.99876	0.99893
70	0.99707	0.99736	0.99764	0.99789	0.99813	0.99836	0.99857	0.99876
80	0.99666	0.99700	0.99731	0.99760	0.99788	0.99813	0.99837	0.99859
90	0.99626	0.99663	0.99698	0.99731	0.99762	0.99791	0.99818	0.99843
100	0.99585	0.99627	0.99666	0.99703	0.99737	0.99769	0.99798	0.99826
110	0.99545	0.99591	0.99634	0.99674	0.99712	0.99747	0.99779	0.99810
120	0.99505	0.99555	0.99602	0.99646	0.99687	0.99725	0.99761	0.99794
130	0.99466	0.99520	0.99571	0.99618	0.99662	0.99703	0.99742	0.99778
140	0.99426	0.99485	0.99539	0.99590	0.99638	0.99682	0.99724	0.99762
150	0.99387	0.99450	0.99508	0.99563	0.99613	0.99661	0.99705	0.99747
160	0.99349	0.99415	0.99477	0.99535	0.99589	0.99640	0.99687	0.99732
170	0.99310	0.99381	0.99447	0.99508	0.99566	0.99619	0.99670	0.99717
180	0.99272	0.99347	0.99416	0.99481	0.99542	0.99599	0.99652	0.99702
190	0.99234	0.99313	0.99386	0.99455	0.99519	0.99579	0.99635	0.99687
200	0.99196	0.99279	0.99356	0.99428	0.99496	0.99559	0.99618	0.99673
210	0.99159	0.99246	0.99327	0.99402	0.99473	0.99539	0.99601	0.99659
220	0.99121	0.99212	0.99297	0399377	0.99450	0.99520	0.99584	0.99645
230	0.99084	0.99180	0.99268	0.99351	0.99428	0.99500	0.99568	0.99631
240	0.99048	0.99147	0.99239	0.99326	0.99406	0.99481	0.99552	0.99617
250	0.99011	0.99115	0.99211	0.99300	0.99384	0.99462	0.99535	0.99604
260	0.98975	0.99082	0.99182	0.99275	0.99362	0.99444	0.99520	0.99591
300	0.98833	0.98957	0.99071	0.99178	0.99278	0.99371	0.99459	0.99540
400	0.98498	0.98660	0.98812	0.98952	0.99084	0.99207	0.99321	0.99428
500	0.98190	0.98391	0.98578	0.98751	0.98913	0.99064	0.99206	0.99337
600	0.97911	0.98149	0.98370	0.98576	0.98767	0.98945	0.99112	0.99268
700	0.97662	0.97935	0.98189	0.98425	0.98645	0.98850	0.99041	0.99219
800	0.97442	0.97750	0.98035	0.98301	0.98547	0.98777	0.98992	0.99192
900	0.97253	0.97593	0.97908	0.98202	0.98474	0.98728	0.98965	0.99185
1000	0.97093	0.97465	0.97809	0.98129	0.98426	0.98702	0.98960	0.99200
1100	0.96965	0.97366	0.97737	0.98081	0.98401	0.98699	0.98977	0.99235
1200	0.96867	0.97295	0.97692	0.98060	0.98402	0.98719	0.99016	0.99291
1300	0.96800	0.97254	0.97674	0.98064	0.98426	0.98762	0.99076	0.99368
1400	0.96763	0.97241	0.97683	0.98093	0.98474	0.98828	0.99157	0.99464
1500	0.96756	0.97256	0.97718	0.98147	0.98545	0.98915	0.99260	0.99580
1600	0.96779	0.97299	0.97780	0.98226	0.98640	0.99024	0.99382	0.99716
1700	0.96832	0.97369	0.97867	0.98329	0.98757	0.99155	0.99525	0.99870
1800	0.96913	0.97466	0.97979	0.98455	0.98896	0.99306	0.99688	1.00043
1900	0.97021	0.97590	0.98116	0.98604	0.99057	0.99478	0.99869	1.00234
2000	0.97157	0.97738	0.98276	0.98775	0.99238	0.99669	1.00069	1.00442
2100	0.97320	0.97912	0.98460	0.98968	0.99440	0.99879	1.00287	1.00668
2200	0.97508	0.98109	0.98666	0.99182	0.99662	1.00108	1.00523	1.00909
2300	0.97720	0.98329	0.98893	0.99416	0.99902	1.00354	1.00775	1.01167
2400	0.97957	0.98571	0.99141	0.99670	1.00161	1.00618	1.01044	1.01440
2500	0.98216	0.98835	0.99410	0.99943	1.00438	1.00899	1.01328	1.01728
2600	0.98497	0.99120	0.99697	1.00233	1.00732	1.01196	1.01627	1.02030
2700	0.98799	0.99423	1.00003	1.00541	1.01042	1.01507	1.01941	1.02345
2800	0.99120	0.99746	1.00327	1.00866	1.01367	1.01834	1.02269	1.02674
2900	0.99461	1.00086	1.00667	1.01206	1.01708	1.02175	1.02610	1.03016
3000	0.99820	1.00444	1.01023	1.01562	1.02063	1.02529	1.02964	1.03369

Table 6-8—Gas Law Deviation Factors for Nitrogen, Calculated from Beattie-Bridgemann Equation of State[14]

P,	Z-Factors for Air at Temperatures (Degrees F) of:							
psia	55	60	65	70	75	80	85	90
0	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000
10	0.99975	0.99977	0.99979	0.99981	0.99983	0.99984	0.99986	0.99987
20	0.99950	0.99954	0.99958	0.99962	0.99965	0.99969	0.99972	0.99975
30	0.99926	0.99932	0.99938	0.99943	0.99948	0.99953	0.99958	0.99963
40	0.99902	0.99910	0.99917	0.99925	0.99932	0.99938	0.99945	0.99951
50	0.99878	0.99888	0.99897	0.99907	0.99915	0.99924	0.99932	0.99940
60	0.99855	0.99867	0.99878	0.99889	0.99899	0.99909	0.99919	0.99928
70	0.99832	0.99845	0.99858	0.99871	0.99883	0.99895	0.99906	0.99917
80	0.99809	0.99824	0.99839	0.99854	0.99868	0.99881	0.99894	0.99906
90	0.99786	0.99804	0.99820	0.99837	0.99852	0.99867	0.99882	0.99896
100	0.99764	0.99783	0.99802	0.99820	0.99837	0.99854	0.99870	0.99885
110	0.99742	0.99763	0.99784	0.99803	0.99822	0.99841	0.99858	0.99875
120	0.99720	0.99743	0.99766	0.99787	0.99808	0.99828	0.99847	0.99866
130	0.99698	0.99724	0.99748	0.99771	0.99793	0.99815	0.99836	0.99856
140	0.99677	0.99704	0.99730	0.99755	0.99779	0.99803	0.99825	0.99847
150	0.99656	0.99685	0.99713	0.99740	0.99766	0.99790	0.99814	0.99837
160	0.99636	0.99667	0.99696	0.99725	0.99752	0.99779	0.99804	0.99829
170	0.99615	0.99648	0.99680	0.99710	0.99739	0.99767	0.99794	0.99820
180	0.99595	0.99630	0.99663	0.99695	0.99726	0.99756	0.99784	0.99812
190	0.99576	0.99612	0.99647	0.99681	0.99713	0.99744	0.99775	0.99804
200	0.99556	0.99595	0.99631	0.99667	0.99701	0.99734	0.99765	0.99796
210	0.99537	0.99577	0.99616	0.99653	0.99689	0.99723	0.99756	0.99788
220	0.99518	0.99560	0.99601	0.99639	0.99677	0.99713	0.99747	0.99781
230	0.99500	0.99544	0.99586	0.99626	0.99665	0.99703	0.99739	0.99774
240	0.99482	0.99527	0.99571	0.99613	0.99654	0.99693	0.99730	0.99767
250	0.99464	0.99511	0.99557	0.99600	0.99643	0.99683	0.99722	0.99760
260	0.99446	0.99495	0.99542	0.99588	0.99632	0.99674	0.99715	0.99754
300	0.99378	0.99435	0.99489	0.99541	0.99591	0.99639	0.99686	0.99731
400	0.99230	0.99304	0.99375	0.99443	0.99508	0.99571	0.99632	0.99691
500	0.99112	0.99202	0.99289	0.99372	0.99453	0.99530	0.99604	0.99675
600	0.99024	0.99130	0.99232	0.99330	0.99424	0.99514	0.99601	0.99685
700	0.98967	0.99088	0.99204	0.99315	0.99422	0.99525	0.99624	0.99720
800	0.98940	0.99074	0.99204	0.99328	0.99447	0.99562	0.99672	0.99779
900	0.98943	0.99091	0.99232	0.99368	0.99499	0.99625	0.99746	0.99862
1000	0.98976	0.99136	0.99289	0.99436	0.99577	0.99713	0.99844	0.99969
1100	0.99039	0.99209	0.99373	0.99530	0.99681	0.99826	0.99966	1.00100
1200	0.99131	0.99311	0.99484	0.99651	0.99811	0.99964	1.00112	1.00254
1300	0.99251	0.99441	0.99623	0.99797	0.99965	1.00126	1.00281	1.00430
1400	0.99399	0.99597	0.99787	0.99969	1.00144	1.00312	1.00474	1.00629
1500	0.99575	0.99780	0.99976	1.00165	1.00346	1.00521	1.00688	1.00849
1600	0.99777	0.99988	1.00191	1.00385	1.00572	1.00751	1.00924	1.01090
1700	1.00005	1.00221	1.00429	1.00628	1.00820	1.01004	1.01181	1.01351
1800	1.00258	1.00479	1.00690	1.00894	1.01089	1.01277	1.01458	1.01631
1900	1.00535	1.00759	1.00974	1.01181	1.01379	1.01570	1.01754	1.01930
2000	1.00834	1.01061	1.01279	1.01489	1.01690	1.01883	1.02069	1.02248
2100	1.01156	1.01385	1.01605	1.01816	1.02019	1.02214	1.02402	1.02582
2200	1.01499	1.01729	1.01950	1.02163	1.02367	1.02563	1.02752	1.02934
2300	1.01862	1.02093	1.02315	1.02528	1.02733	1.02930	1.03119	1.03301
2400	1.02244	1.02475	1.02697	1.02910	1.03115	1.03312	1.03501	1.03684
2500	1.02645	1.02875	1.03096	1.03309	1.03513	1.03710	1.03899	1.04081
2600	1.03063	1.03292	1.03512	1.03724	1.03927	1.04123	1.04311	1.04492
2700	1.03497	1.03725	1.03943	1.04153	1.04355	1.04550	1.04736	1.04916
2800	1.03947	1.04173	1.04389	1.04597	1.04797	1.04990	1.05175	1.05353
2900	1.04412	1.04635	1.07849	1.05054	1.05252	1.05443	1.05626	1.05802
3000	1.04891	1.05110	1.05321	1.05524	1.05720	1.05907	1.06088	1.06262

P,	Z-Factors for Air at Temperatures (Degrees C) of:							
psia	0	5	10	15	20	25	30	35
0	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000
10	0.99965	0.99969	0.99973	0.99977	0.99980	0.99983	0.99986	0.99989
20	0.99930	0.99939	0.99946	0.99954	0.99960	0.99967	0.99973	0.99978
30	0.99896	0.99908	0.99920	0.99931	0.99941	0.99950	0.99959	0.99968
40	0.99862	0.99878	0.99894	0.99908	0.99922	0.99934	0.99946	0.99957
50	0.99828	0.99849	0.99868	0.99886	0.99903	0.99919	0.99933	0.99947
60	0.99795	0.99819	0.99843	0.99864	0.99884	0.99903	0.99921	0.99937
70	0.99761	0.99790	0.99817	0.99843	0.99866	0.99888	0.99909	0.99928
80	0.99729	0.99762	0.99792	0.99821	0.99848	0.99873	0.99896	0.99918
90	0.99696	0.99733	0.99768	0.99800	0.99830	0.99858	0.99885	0.99909
100	0.99664	0.99705	0.99744	0.99779	0.99813	0.99844	0.99873	0.99900
110	0.99632	0.99677	0.99719	0.99759	0.99795	0.99830	0.99862	0.99892
120	0.99601	0.99650	0.99696	0.99739	0.99779	0.99816	0.99851	0.99883
130	0.99569	0.99623	0.99672	0.99719	0.99762	0.99802	0.99840	0.99875
140	0.99538	0.99596	0.99649	0.99699	0.99745	0.99789	0.99829	0.99867
150	0.99508	0.99569	0.99626	0.99680	0.99729	0.99776	0.99819	0.99860
160	0.99478	0.99543	0.99604	0.99660	0.99713	0.99763	0.99809	0.99852
170	0.99448	0.99517	0.99582	0.99642	0.99698	0.99750	0.99799	0.99845
180	0.99418	0.99491	0.99560	0.99623	0.99683	0.99738	0.99790	0.99838
190	0.99389	0.99466	0.99538	0.99605	0.99667	0.99726	0.99780	0.99831
200	0.99360	0.99441	0.99517	0.99587	0.99653	0.99714	0.99771	0.99825
210	0.99331	0.99416	0.99496	0.99569	0.99638	0.99703	0.99763	0.99819
220	0.99303	0.99392	0.99475	0.99552	0.99624	0.99691	0.99754	0.99813
230	0.99275	0.99368	0.99454	0.99535	0.99610	0.99680	0.99746	0.99807
240	0.99247	0.99344	0.99434	0.99518	0.99596	0.99669	0.99738	0.99802
250	0.99220	0.99321	0.99414	0.99502	0.99583	0.99659	0.99730	0.99796
260	0.99193	0.99298	0.99395	0.99485	0.99570	0.99649	0.99722	0.99791
300	0.99089	0.99208	0.99320	0.99424	0.99520	0.99611	0.99695	0.99774
400	0.98851	0.99008	0.99154	0.99289	0.99416	0.99534	0.99644	0.99747
500	0.98647	0.98839	0.99018	0.99185	0.99339	0.99484	0.99618	0.99744
600	0.98478	0.98704	0.98914	0.99109	0.99291	0.99460	0.99618	0.99766
700	0.98343	0.98602	0.98841	0.99064	0.99271	0.99464	0.99644	0.99811
800	0.98244	0.98532	0.98800	0.99048	0.99279	0.99494	0.99694	0.99881
900	0.98180	0.98496	0.98789	0.99061	0.99315	0.99550	0.99769	0.99974
1000	0.98151	0.98493	0.98810	0.99104	0.99378	0.99632	0.99869	1.00090
1100	0.98156	0.98522	0.98861	0.99176	0.99468	0.99740	0.99993	1.00229
1200	0.98196	0.98583	0.98942	0.99276	0.99585	0.99873	1.00141	1.00390
1300	0.98270	0.98676	0.99053	0.99403	0.99728	1.00030	1.00312	1.00574
1400	0.98376	0.98800	0.99193	0.99558	0.99897	1.00212	1.00505	1.00778
1500	0.98515	0.98955	0.99362	0.99740	1.00091	1.00417	1.00721	1.01004
1600	0.98686	0.99138	0.99558	0.99947	1.00308	1.00645	1.00958	1.01249
1700	0.98886	0.99350	0.99780	1.00179	1.00550	1.00894	1.01215	1.01514
1800	0.99117	0.99589	1.00028	1.00435	1.00813	1.01165	1.01493	1.01798
1900	0.99375	0.99855	1.00301	1.00715	1.01099	1.01457	1.01790	1.02100
2000	0.99661	1.00147	1.00598	1.01017	1.01406	1.01768	1.02105	1.02419
2100	0.99973	1.00463	1.00918	1.01340	1.01733	1.02098	1.02439	1.02756
2200	1.00310	1.00802	1.01259	1.01684	1.02079	1.02447	1.02789	1.03108
2300	1.00670	1.01163	1.01622	1.02047	1.02444	1.02812	1.03156	1.03476
2400	1.01053	1.01546	1.02004	1.02430	1.02826	1.03195	1.03538	1.03859
2500	1.01458	1.01949	1.02405	1.02830	1.03225	1.03593	1.03936	1.04256
2600	1.01882	1.02371	1.02824	1.03247	1.03640	1.04006	1.04348	1.04666
2700	1.02326	1.02371	1.00824	1.03247	1.03640	1.04006	1.04348	1.04666
2800	1.02788	1.03267	1.03713	1.04128	1.04515	1.04875	1.05211	1.05524
2900	1.03267	1.03740	1.04181	1.04591	1.04973	1.05329	1.05661	1.05971
3000	1.03762	104228	1.04663	1.05067	1.05444	1.05796	1.06123	1.06429

Table 6-9—Gas Law Deviation Factors for Helium, Calculated from Beattie-Bridgemann Equation of State[14]

P,	Z-Factors for Air at Temperatures (Degrees F) of:								P,	Z-Factors for Air at Temperatures (Degrees C) of:							
psia	55	60	65	70	75	80	85	90	psia	0	5	10	15	20	25	30	35
0	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000	0	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000
10	1.00038	1.00038	1.00037	1.00037	1.00037	1.00036	1.00036	1.00036	10	1.00040	1.00039	1.00038	1.00038	1.00037	1.00036	1.00036	1.00035
20	1.00076	1.00075	1.00074	1.00074	1.00073	1.00073	1.00072	1.00071	20	1.00079	1.00078	1.00077	1.00075	1.00074	1.00073	1.00072	1.00071
30	1.00114	1.00113	1.00112	1.00111	1.00110	1.00109	1.00108	1.00107	30	1.00119	1.00117	1.00115	1.00113	1.00111	1.00109	1.00108	1.00106
40	1.00152	1.00150	1.00149	1.00148	1.00146	1.00145	1.00144	1.00143	40	1.00158	1.00155	1.00153	1.00150	1.00148	1.00146	1.00144	1.00141
50	1.00189	1.00188	1.00186	1.00184	1.00183	1.00181	1.00180	1.00178	50	1.00198	1.00194	1.00191	1.00188	1.00185	1.00182	1.00179	1.00177
60	1.00227	1.00225	1.00223	1.00221	1.00219	1.00217	1.00216	1.00214	60	1.00237	1.00233	1.00229	1.00226	1.00222	1.00219	1.00215	1.00212
70	1.00265	1.00263	1.00260	1.00258	1.00256	1.00254	1.00251	1.00249	70	1.00276	1.00272	1.00267	1.00263	1.00259	1.00255	1.00251	1.00247
80	1.00303	1.00303	1.00297	1.00295	1.00292	1.00290	1.00287	1.00285	80	1.00316	1.00311	1.00306	1.00301	1.00296	1.00291	1.00287	1.00282
90	1.00341	1.00338	1.00335	1.00332	1.00329	1.00326	1.00323	1.00320	90	1.00355	1.00349	1.00344	1.00338	1.00333	1.00328	1.00323	1.00318
100	1.00378	1.00375	1.00372	1.00368	1.00365	1.00362	1.00359	1.00356	100	1.00395	1.00388	1.00382	1.00376	1.00370	1.00364	1.00358	1.00353
110	1.00416	1.00412	1.00409	1.00405	1.00402	1.00398	1.00.95	1.00391	110	1.00434	1.00427	1.00420	1.00413	1.00406	1.00400	1.00394	1.00388
120	1.00454	1.00450	1.00446	1.00442	1.00438	1.00434	1.00430	1.00427	120	1.00473	1.00465	1.00458	1.00450	1.00443	1.00436	1.00430	1.00423
130	1.00491	1.00487	1.00483	1.00478	1.00474	1.00470	1.00466	1.00462	130	1.00513	1.00504	1.00496	1.00488	1.00480	1.00473	1.00465	1.00458
140	1.00529	1.00524	1.00520	1.00515	1.00511	1.00506	1.00502	1.00498	140	1.00552	1.00543	1.00534	1.00525	1.00517	1.00509	1.00501	1.00493
150	1.00567	1.00562	1.00557	1.00552	1.00547	1.00542	1.00538	1.00533	150	1.00591	1.00581	1.00572	1.00563	1.00554	1.00545	1.00537	1.00529
160	1.00604	1.00599	1.00594	1.00588	1.00583	1.00578	1.00573	1.00568	160	1.00630	1.00620	1.00610	1.00600	1.00591	1.00581	1.00572	1.00564
170	1.00642	1.00636	1.00631	1.00625	1.00620	1.00614	1.00609	1.00604	170	1.00670	1.00659	1.00648	1.00637	1.00627	1.00617	1.00608	1.00599
180	1.00680	1.00673	1.00668	1.00662	1.00656	1.00650	1.00645	1.00639	180	1.00709	1.00697	1.00686	1.00675	1.00664	1.00654	1.00644	1.00634
190	1.00717	1.00711	1.00704	1.00698	1.00692	1.00686	1.00680	1.00675	190	1.00748	1.00736	1.00724	1.00712	1.00701	1.00690	1.00679	1.00669
200	1.00755	1.00748	1.00741	1.00735	1.00728	1.00722	1.00716	1.00710	200	1.00787	1.00774	1.00762	1.00749	1.00737	1.00726	1.00715	1.00704
210	1.00792	1.00785	1.00778	1.00771	1.00765	1.00758	1.00752	1.00745	210	1.00826	1.00813	1.00799	1.00787	1.00774	1.00762	1.00750	1.00739
220	1.00830	1.00822	1.00815	1.00808	1.00801	1.00794	1.00787	1.00781	220	1.00866	1.00851	1.00837	1.00824	1.00811	1.00798	1.00786	1.00774
230	1.00867	1.00859	1.00852	1.00844	1.00837	1.00830	1.00823	1.00816	230	1.00905	1.00890	1.00875	1.00861	1.00847	1.00834	1.00821	1.00809
240	1.00905	1.00897	1.00889	1.00881	1.00873	1.00866	1.00858	1.00851	240	1.00944	1.00928	1.00913	1.00898	1.00884	1.00870	1.00857	1.00844
250	1.00942	1.00934	1.00925	1.00917	1.00909	1.00902	1.00894	1.00886	250	1.00983	1.00967	1.00951	1.00935	1.00921	1.00906	1.00892	1.00879
260	1.00980	1.00971	1.00962	1.00954	1.00946	1.00937	1.00929	1.00922	260	1.01022	1.01005	1.00988	1.00973	1.00957	1.00942	1.00928	1.00914
300	1.01129	1.01119	1.01109	1.01099	1.01090	1.01081	1.01071	1.01062	300	1.01178	1.01158	1.01139	1.01121	1.01103	1.01086	1.01069	1.01053
400	1.01502	1.01488	1.01475	1.01462	1.01450	1.01437	1.01425	1.01413	400	1.01567	1.01541	1.01515	1.01491	1.01468	1.01445	1.01423	1.01401
500	1.01873	1.01856	1.01840	1.01824	1.01808	1.01792	1.01777	1.01762	500	1.01953	1.01921	1.01890	1.01859	1.01830	1.01801	1.01774	1.01747
600	1.02242	1.02222	1.02202	1.02183	1.02164	1.02145	1.02127	1.02109	600	1.02338	1.02229	1.02262	1.02226	1.02191	1.02157	1.02124	1.02092
700	1.02609	1.02586	1.02563	1.02541	1.02519	1.02497	1.02476	1.02455	700	1.02721	1.02676	1.02633	1.02590	1.02550	1.02510	1.02472	1.02434
800	1.02974	1.02948	1.02922	1.02897	1.02872	1.02847	1.02823	1.02799	800	1.03102	1.03051	1.03001	1.02953	1.02907	1.02862	1.02818	1.02776
900	1.03338	1.03309	1.03280	1.03251	1.03223	1.03195	1.03168	1.03142	900	1.03482	1.03424	1.03368	1.03315	1.03262	1.03212	1.03163	1.03115
1000	1.03700	1.03668	1.03635	1.03604	1.03573	1.03542	1.03512	1.03483	1000	1.03859	1.03795	1.03734	1.03674	1.03616	1.03560	1.03506	1.03454
1100	1.04061	1.04025	1.03990	1.03955	1.03921	1.03887	1.03854	1.03822	1100	1.04235	1.04165	1.04097	1.04032	1.03969	1.03907	1.03848	1.03790
1200	1.04420	1.04381	1.04342	1.04305	1.04267	1.04231	1.04195	1.04160	1200	1.04609	1.04533	1.04460	1.04388	1.04320	1.04253	1.04188	1.04126
1300	1.04777	1.04735	1.04693	1.04653	1.04613	1.04573	1.04535	1.04497	1300	1.04981	1.04899	1.04820	1.04743	1.04669	1.04597	1.04527	1.04459
1400	1.05133	1.05087	1.05043	1.04999	1.04956	1.04914	1.04872	1.04832	1400	1.05352	1.05264	1.05179	1.05096	1.05016	1.04939	1.04864	1.04792
1500	1.05487	1.05438	1.05391	1.05344	1.05298	1.05253	1.05209	1.05165	1500	1.05721	1.05627	1.05536	1.05448	1.05363	1.05280	1.05200	1.05122
1600	1.05839	1.05788	1.05737	1.05688	1.05639	1.05591	1.05544	1.05498	1600	1.06089	1.05988	1.05892	1.05798	1.05707	1.05620	1.05534	1.05452
1700	1.06190	1.06136	1.06082	1.06030	1.05978	1.05927	1.05877	1.05828	1700	1.06454	1.06348	1.06246	1.06147	1.06051	1.05958	1.05867	1.05780
1800	1.06540	1.06482	1.06426	1.06370	1.06316	1.06262	1.06210	1.06158	1800	1.06819	1.06707	1.06599	1.06494	1.06392	1.06294	1.06199	1.06107
1900	1.06888	1.06827	1.06768	1.06710	1.06652	1.06596	1.06540	1.06486	1900	1.07181	1.07064	1.06950	1.06839	1.06733	1.06629	1.06529	1.06432
2000	1.07235	1.07171	1.07109	1.07047	1.06987	1.06928	1.06870	1.06813	2000	1.07543	1.07419	1.07299	1.07184	1.07072	1.06963	1.06858	1.06756
2100	1.07580	1.07513	1.07448	1.07384	1.07321	1.07259	1.07198	1.07138	2100	1.07902	1.07773	1.07648	1.07527	1.07409	1.07296	1.07186	1.07079
2200	1.07924	1.07854	1.07786	1.07719	1.07653	1.07588	1.07525	1.07462	2200	1.08260	1.08125	1.07994	1.07868	1.07746	1.07627	1.07512	1.07401
2300	1.08266	1.08194	1.08123	1.08053	1.07984	1.07917	1.07850	1.07785	2300	1.08617	1.08476	1.08340	1.08208	1.08080	1.07957	1.07837	1.07721
2400	1.08607	1.08532	1.08458	1.08385	1.08314	1.08243	1.08174	1.08107	2400	1.08972	1.08826	1.08684	1.08547	1.08414	1.08285	1.08161	1.08040
2500	1.08947	1.08869	1.08792	1.08716	1.08642	1.08569	1.08497	1.08427	2500	1.09326	1.09174	1.09027	1.08884	1.08746	1.08613	1.08483	1.08358
2600	1.09285	1.09204	1.09124	1.09046	1.08969	1.08893	1.08819	1.08746	2600	1.09679	1.09521	1.09368	1.09220	1.09077	1.08939	1.08804	1.08674
2700	1.09622	1.09538	1.09456	1.09375	1.09295	1.09217	1.09140	1.09064	2700	1.10030	1.09866	1.09708	1.09555	1.09407	1.09263	1.09124	1.08990
2800	1.09958	1.09871	1.09786	1.09702	1.09619	1.09538	1.09459	1.09381	2800	1.10379	1.10210	1.10047	1.09888	1.09735	1.09587	1.09443	1.09304
2900	1.10293	1.10203	1.10114	1.10028	1.09943	1.09859	1.09777	1.09696	2900	1.10728	1.10553	1.10384	1.10221	1.10062	1.09909	1.09761	1.09617
3000	1.10626	1.10533	1.10442	1.10353	1.10265	1.10179	1.10094	1.10010	3000	1.11075	1.10894	1.10720	1.10551	1.10388	1.10230	1.10077	1.09929

T = absolute temperature of the gas during the measurement of b, degrees K.

M = gas molecular weight.

c = dimensionless "acceptance coefficient," which is slightly less than 1.0, according to Klinkenberg.[3]

r = radius of randomly distributed (spatially) capillaries that approximate a porous medium in Klinkenberg's model, μm.

In Equation 12, the factor in brackets depends upon gas properties only, and the factor in parentheses is rock dependent. If we define the rock-dependent factor, r/c, as the rock's "effective gas slippage radius," r_b, and solve for it by rearranging Equation 12:

$$r_b = \frac{99.5\mu}{b}\sqrt{\frac{T}{M}}, \tag{13}$$

then this is the recommended gas slippage factor to be reported, which is independent of the gas used and temperature. It should be reported in addition to b, or instead of it.

Klinkenberg's randomly distributed capillary analogy relates r_b to permeability and porosity by:

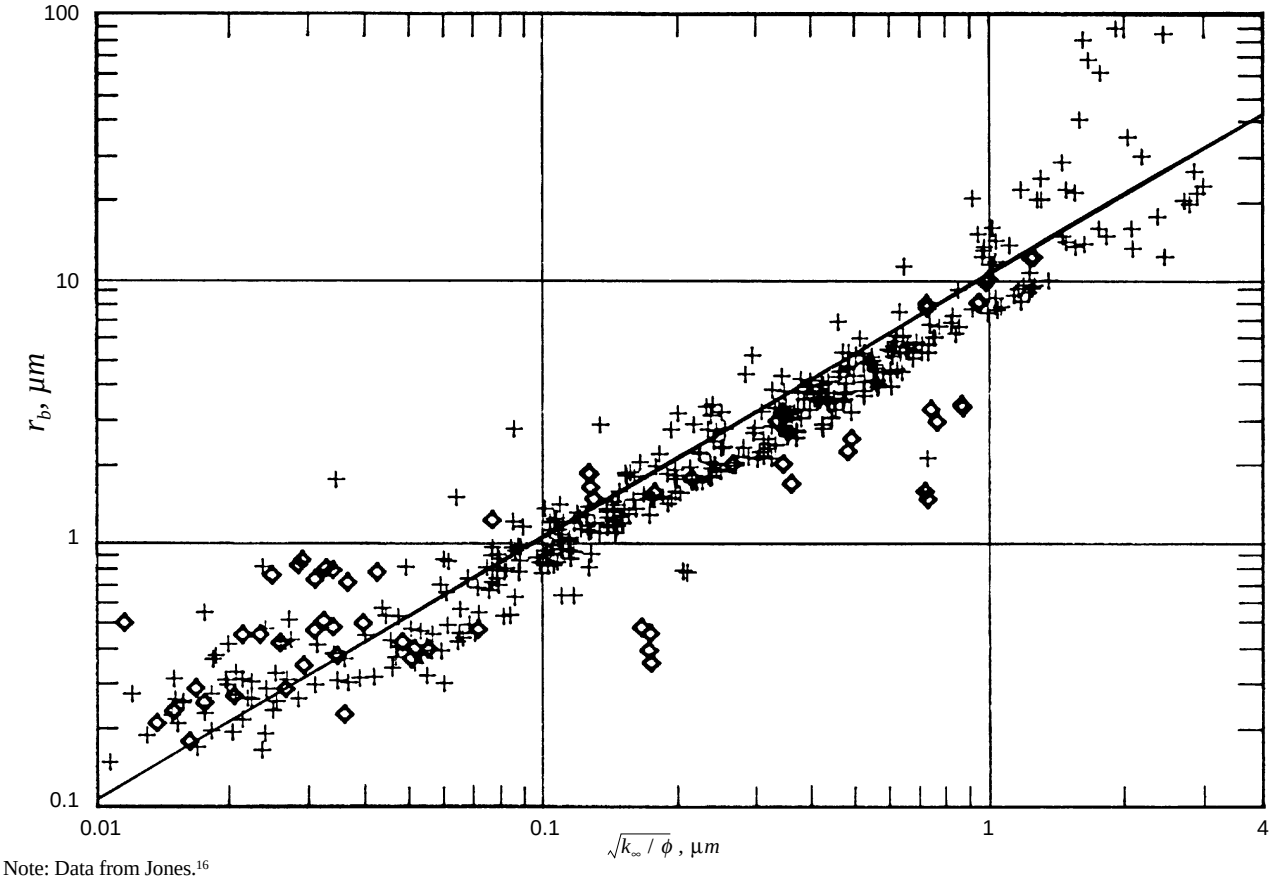
$$r_b = C_b\sqrt{\frac{k_\infty}{\phi}} \tag{14}$$

where k_∞ is Klinkenberg permeability, (μm^2), and ϕ is porosity (fraction). In Klinkenberg's model, C_b is equal to $\sqrt{24}/c$, or approximately 5. If we relax this "too simple" model, C_b can be found empirically. In Figure 6-2, r_b is plotted vs. $\sqrt{k_\infty/\phi}$. The slope of the line drawn in this log-log plot is unity. From its position, C_b is calculated to be approximately 11. The data, obtained using helium in several core plugs, show considerable scatter. Except for value of the constant, they qualitatively support Klinkenberg's model. The gas slippage parameters, b or r_b, are anticipated to be functions of pore structure, which may account for some of the scatter.

6.2.1.2 Steady-State Flow of Gas—The Forchheimer Equation

Steady state is achieved when the upstream and downstream pressures and flow rate all become invariant with time. At steady state, mass flow rate is constant throughout the sample and does not change with time. Therefore:

$$\rho q_s = \rho_r q_r \tag{15}$$



Note: Data from Jones.[16]

Figure 6-2—Rock-Dependent Gas Slippage Radius, r_b, as a Function of Permeability and Porosity

The subscript r refers to reference conditions of temperature and pressure under which the mass flow rate is measured. Thus at steady-state, isothermal conditions, where the reference temperature is the same as the flowing gas temperature:

$$\frac{Pq_s}{z} = \frac{P_r q_r}{z_r} \tag{16}$$

Substituting Equations 7, 9, and 16 into Equation 5 yields the differential form of the Forchheimer equation for isothermal, steady-state flow of gases, corrected for gas slippage:

$$C_1\left(\frac{-dP}{ds}\right) = \frac{C_2 \mu P_r q_r z}{A k_\infty z_t (P+b)} + \frac{C_3 \beta M P_r^{\,2} q_r^{\,2} z}{A^2 R T z_r^{\,2} P} \tag{17}$$

The integrated form of this equation can be used to obtain k_∞, b, and β. This requires a minimum of three, and preferably at least six measurements in which the upstream pressure (and/or optionally the downstream pressure) is varied widely to cause substantial variations in the mean pore pressure and flow velocity. For routine measurements, this is not practicable (cost-wise) under steady-state conditions. However, for most cases, adequate information for the calculation of these parameters can be obtained from a single pressure-falloff run (see 6.4.1.1 and 6.4.1.2).

6.2.1.3 The Slip-Corrected Darcy Equation

Whenever its last term becomes negligibly small, Equation 17 reduces to the differential form of Darcy's Law, corrected for gas slippage, and is the basis for most practical, routine measurements. Small pressure gradients lead to small flow velocities, that are squared in the last term, and are therefore small. The term's magnitude also depends on the values of β and gas density. Density is a function of the temperature, pressure, and molecular weight of the gas used.

Figure 6-3 shows typical values of β as a function of permeability for sandstone (open circles) and carbonate rocks (squares) (data from Jones[16]), and for homogeneous unconsolidated sands (solid circles, data from Geertsma[17]). The considerable scatter is believed to be related to heterogeneity in the rock.[16] Samples with fractures or thin layers of higher permeability material parallel to the flow direction have higher β's than homogeneous rock, due to locally high velocities in these high permeability "streaks." Figure 6-3 contains two dashed curves that encompass most of the points, and a central "best fit" curve.

Figure 6-4, which is based on the data in Figure 6-3, presents maximum allowable overall pressure gradients, $\Delta p/L$, that are sufficiently small to permit use of the slip-corrected Darcy equation for the calculation of permeability without incurring significant error due to inertial resistance. The curves shown are for air at 72°F, and for downstream pressures of 14.7, 50, 100, and 200 psia. Because of the scatter in β, these curves are approximate, and are to be interpreted as follows: If the actual β for a particular sample happens to lie on the upper dashed line in Figure 6-3, and the maximum $\Delta p/L$ as shown in Figure 6-4, for the conditions used, were applied for the permeability measurement, then the calculated permeability would be 5 percent low if the final term in Equation 17 were ignored. If the actual β were twice the value shown on the upper dashed line of Figure 6-3, and the same $\Delta p/L$ were applied, then the calculated permeability would be 10 percent too low. On the other hand, if the actual β were a factor of 10 smaller than the one indicated by the upper dashed line (thus, is closer to the high density of points), the permeability error would be only 0.5 percent, etc. Use of the maximum $\Delta p/L$ values shown in Figure 6-4 will result in errors (due to inertial resistance) of less than 2 percent for most samples when the slip-corrected Darcy equation is used for the calculation of permeability.

Starting with the highest permeabilities for the 14.7 psia downstream pressure in Figure 6-4, the maximum allowable pressure drop increases with decreasing permeability, as expected. However, for permeabilities below about 0.3 millidarcys, $\Delta p_{max}/L$ decreases with decreasing permeability. This unexpected result is a consequence of gas slippage. Slippage is greatest at the lowest permeabilities, causing higher gas flow rates than would occur without slippage.

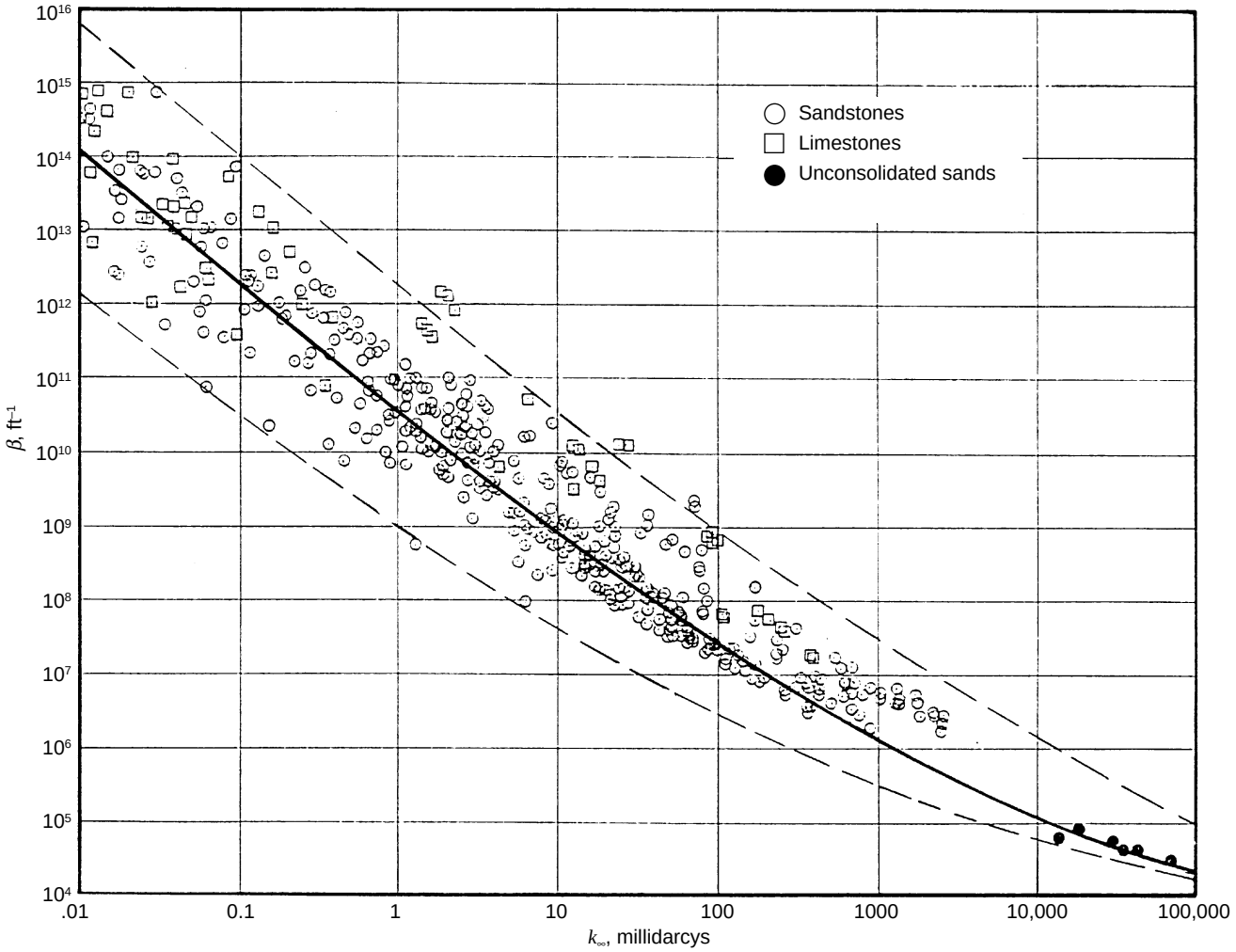
Except for very low permeabilities, the maximum allowable overall gradient is reduced as downstream pressure is increased. Increased back pressure increases gas density, thereby increasing the significance of the final term in Equation 17. This result should be acknowledged when making multiple measurements with increasingly high pore pressures for the Klinkenberg gas slippage correction. Apparent gas permeabilities measured at high pore pressures can be too low due to the inadvertent introduction of non-negligible inertial resistance.

Maximum $\Delta p/L$ values are gas specific. Values for nitrogen and air are similar, but those for helium are higher. Helium's viscosity is higher, and its molecular weight is less than that of air. Equations for calculating $\Delta p/L$ for other gases and liquids are developed in A.6.8.1.

The condition of Stokes flow is met when the last term of Equation 17 is negligible. The remaining slip-corrected Darcy equation can be integrated and used for calculation of Klinkenberg permeabilities for several frequently used flow configurations:

$$k_\infty = \frac{2 C_2 \mu P_r q_r z_m}{C_1 z_r G_f (P_1 - P_2)(P_1 + P_2 + 2b)} \tag{18}$$

Where C_1 and C_2 are conversion constants from Table 6-1 to allow for various sets of units for the variables. P_1 and P_2 are the injection and outflow absolute gas pressures, respectively.



Note: Data from Jones[16], and Geertsma[17].

Figure 6-3—Inertial Resistivity, β, as a Function of Permeability

To perform this integration, it was assumed that z in Equation 17 is replaceable by its mean value, z_m. Gas deviation factors z_m and z_r are calculated at the mean pore pressure, P_m (see Equation 20, below), and the absolute reference pressure, P_r, at which q_r is measured, respectively, and at the temperature of the gas. G_f is a geometric factor, which has the dimension of length. The particular factor for each of several flow configurations is given in sections following 6.3.1. The Klinkenberg gas slippage factor, b, must be determined from multiple measurements, or from a correlation. If it is obtained from a correlation, this fact shall be clearly stated.

To obtain b, from which r_b is calculated using Equation 13, the apparent gas permeability, k_g, is measured at a minimum of three (and preferably at least four) different mean pore pressures. Each permeability is calculated from:

$$k_g = \frac{2C_2\mu P_r q_r z_m}{C_1 z_r G_f (P_1 - P_2)(P_1 + P_2)} \tag{19}$$

and is plotted against the reciprocal of the mean pore pressure as illustrated by Figure 6-5. Mean pore pressure is defined as:

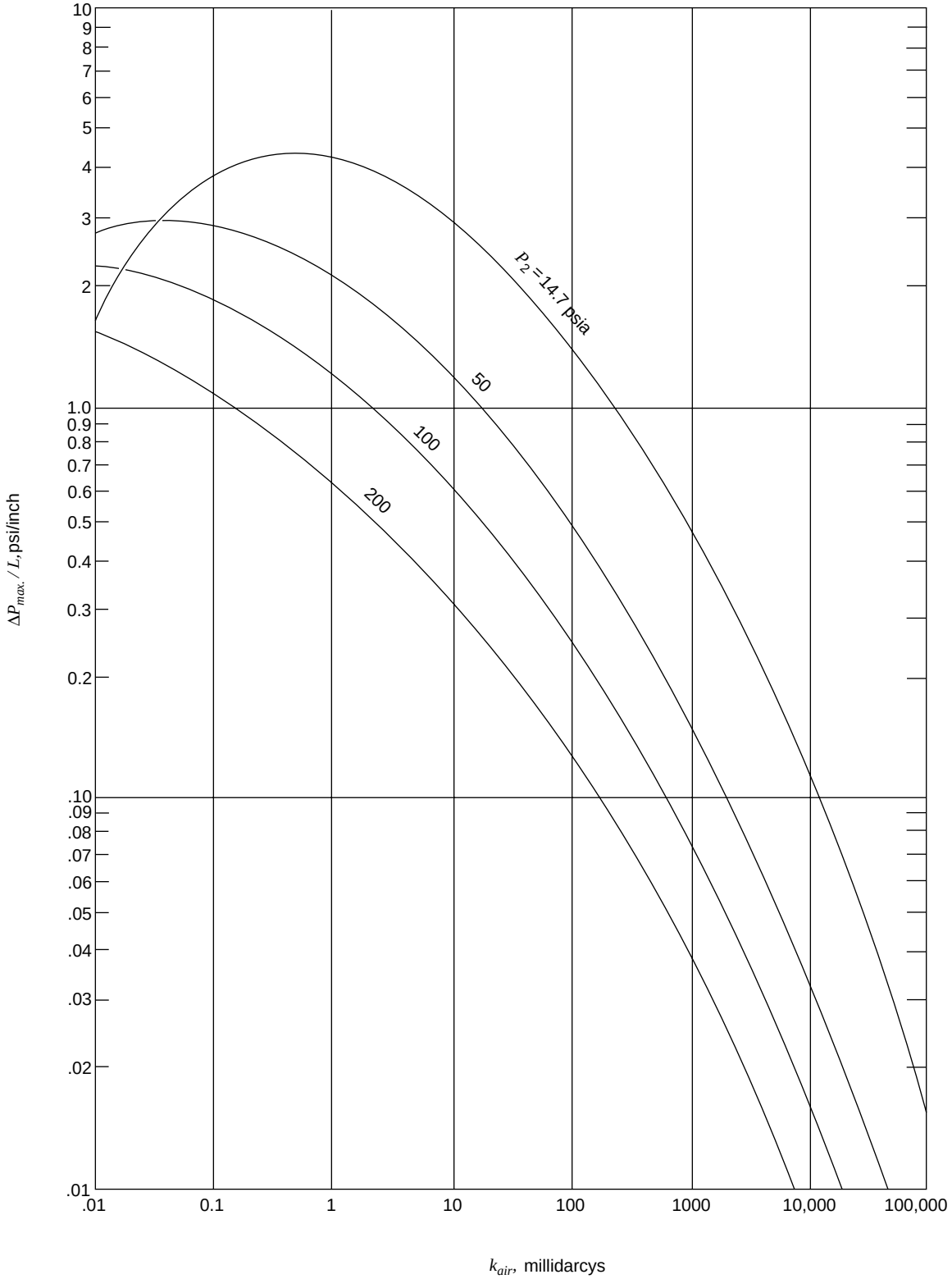
$$P_m = \tfrac{1}{2}(P_1 + P_2) \tag{20}$$

The intercept of such a plot is k_∞, and its slope is equal to bk_∞, in accordance with the Klinkenberg relationship:

$$k_g = k_\infty\left(1 + \frac{b}{P_m}\right) \tag{21}$$

The slope and intercept can be found by linear regression. It is essential to ensure that inertial resistance is negligible for each measurement. Otherwise, k_g will be low, especially at higher P_m values. Departure from linearity provides a self check on consistency.

Often, for routine permeability measurements, only gas permeabilities (uncorrected for gas slippage), as calculated from Equation 19, are reported. For these measurements, it is



Note: For air at 72°F.

Figure 6-4—Maximum Allowable Overall Pressure Gradients for Use of Slip-Corrected Darcy Equation

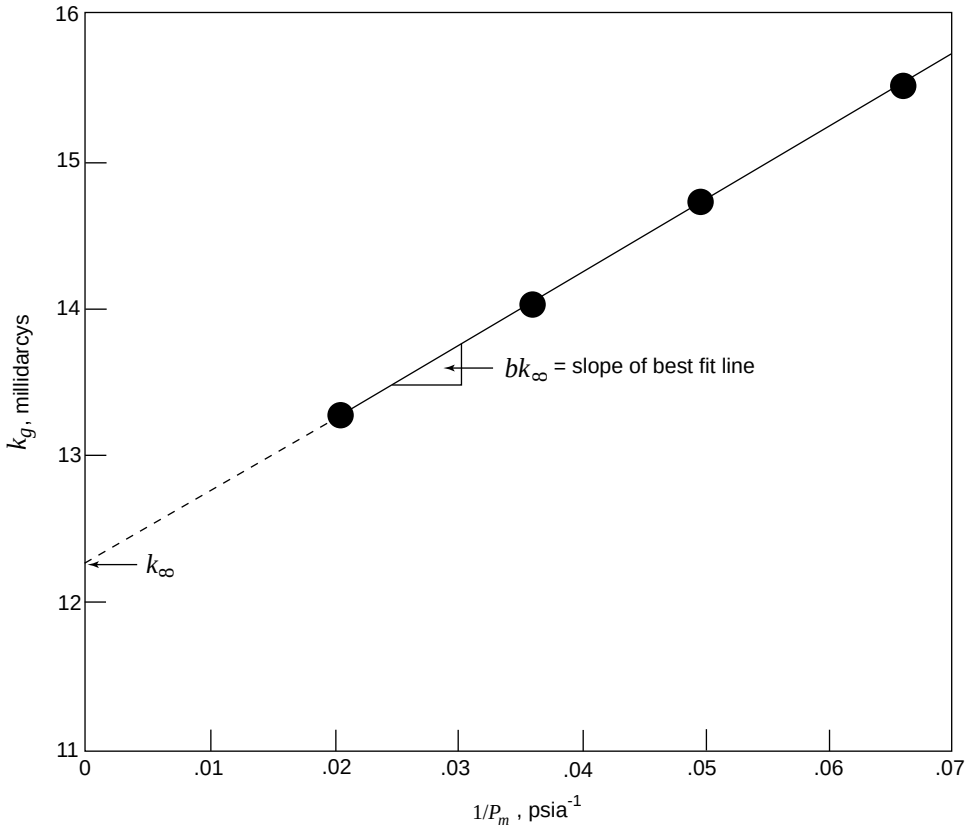


Figure 6-5—Type of Plot Used To Obtain Klinkenberg Gas Slippage Factor, *b*

essential to report the gas used and its mean pore pressure. Otherwise, the reported permeabilities are not fully specified.

6.2.2 Steady-State Permeability Equations For Liquids

Liquids have lower compressibilities, higher densities, and higher viscosities than gases. For steady-state measurements involving small pressure differences, liquids can be treated as incompressible fluids. Due to the higher liquid densities, gravity effects normally cannot be ignored in permeability measurements, except for horizontal flow. Departure from Darcy's Law is rarely a problem with liquids. Their higher viscosities result in significantly lower volumetric fluxes than observed for gases at the same pressure gradients. But their higher densities somewhat offset the viscosity effect with regard to inertial resistance. See A.6.8.1 for the calculation of maximum allowable $\Delta p/L$ values to be used for liquid permeability measurements when Darcy's Law is used.

The mean free path between flowing liquid molecules is so small that slippage is not observed. However, liquids—especially aqueous solutions—can react with constituents of a porous rock, altering its permeability. Also, care must be taken to avoid mobilizing fines in a rock at high liquid fluxes.

Darcy's Law, including gravity effects, for Stokes flow of liquids through permeable media may be stated in general terms as:

$$v_s = \frac{q}{A} = \frac{-C_1 k}{C_2 \mu}\left(\frac{dp}{ds} - \frac{pg}{C_4}\frac{dz}{ds}\right) \qquad (22)$$

Where:

s = distance along the direction of flow.

v_s = volumetric flux (volume of flow through a unit area of the porous medium per unit time).

z = vertical coordinate (increasing downward).

ρ = density of the liquid.

$\frac{dp}{ds}$ = pressure gradient along s at the point to which refers.

μ = viscosity of the liquid.

k = permeability of the medium.

q = volume rate of flow.

A = cross-sectional area perpendicular to lines of flow.

C_1, C_2, and C_4 are constants, found in Table 6-1, that make the units consistent.

6.2.2.1 Special Cases for Steady-State Flow of Incompressible Liquids

Equation 22 may be reduced to simpler forms under special conditions of flow. Some of the more important of these are as follows.

6.2.2.1.1 Horizontal Flow of Liquids

Because there is no vertical component of flow, $dz/ds = 0$, and integration of Equation 22 results in:

$$k = \frac{C_2 q \mu}{C_1 G_f (p_1 - p_2)} \tag{23}$$

Where G_f is a geometrical factor, which has the dimension of length. Factors for axial, transverse, or radial flow are given by Equations 27, 29, or 30, respectively, following 6.3.1.

6.2.2.1.2 Vertical Flow of Liquids

Two sub-cases arise, depending whether the flow is downward or upward, and dz/ds is equal to +1 or -1, respectively:

a. Downward flow with a driving head h (as defined by Figure 6-6):

$$k = \frac{C_2 C_4 q \mu L}{C_1 A \rho g (h + L)} \tag{24}$$

where L is the length of the sample and h is the height of the driving head. If the driving head, h, is zero, we have free downward flow, in which flow rate is independent of sample length.

b. Upward flow with driving head, h (as defined by Figure 6-7):

$$k = \frac{C_2 C_4 q \mu L}{C_1 A \rho g h} \tag{25}$$

If upward steady-state flow is achieved with an imposed pressure difference, Δp, which is measured with a differential pressure transducer with both ports at the same elevation (as shown in Figure 6-8) and both transducer lines completely filled with the same liquid as the flowing liquid, then:

$$k = \frac{C_2 q \mu L}{C_1 A \Delta p} \tag{26}$$

After steady-state is achieved (i.e., the Δp and flow rate are invariant with time), the inlet and outlet valves should be closed simultaneously, and the differential pressure transducer allowed to reach equilibrium. The equilibrium reading should be zero. If it is not zero, this reading should be subtracted from the steady-state reading.

6.2.3 Permeability Decrease At Elevated Net Stresses

The permeability of reservoir rock decreases with increasing net stress. Net stress for permeability is usually defined as the difference between confining stress and mean pore pressure. Figure 6-9 illustrates typical permeability reduction with increasing net hydrostatic stress. Percentage reductions are usually greater with lower permeability plugs. For example, zero-stress permeabilities of plugs 6-2, 8-2, and 11-2 in Figure 6-9 are about 700, 40, and 10 millidarcys, respectively. The permeability of a tight gas sand (e.g., 0.01 millidarcy or less at zero stress) can be reduced to 10 percent or less of its zero-stress permeability by high net compressive stresses.

6.3 PRACTICAL APPLICATIONS FOR STEADY-STATE PERMEABILITY MEASUREMENTS

Several practical configurations for routine, steady-state measurement of permeability are described, first for gases, then for liquids. Each may have particular advantages and limitations (e.g., speed; cost; accuracy; permeability range that can be accommodated; stress levels achievable; directional measurements; susceptibility to error; etc.) that are discussed. Certain precautions to avoid major pitfalls are presented.

Even though we ultimately calculate the permeability of a sample, which is a measure of its fluid conductivity, it is the conductance of the sample that dictates its fluid flow behavior. Conductance depends upon path length and area available for flow, as well as the permeability of the sample. Therefore, the measurable permeability range can be extended in some flow configurations by judicious choice of sample dimensions. For example, to maintain reasonable flow rates with axial flow when permeabilities are very high, long, small-diameter samples can be prepared. Conversely, for low permeability samples, short, larger-diameter plugs are desirable. Other conditions being equal, the maximum permeability that can be measured accurately with a three-inch-long plug is three times as large as can be measured on an otherwise identical one-inch-long plug.

Samples submitted for measurement often range in permeability from less than 0.001 to more than 30,000 millidarcys—a ratio of more than 3×10^7. To accommodate this variation, multiple ranges of pressure and flow rate measuring and/or controlling devices must either be insertable into a permeameter, or selectable from multiple devices that are built in. For simplicity, only a single set of devices is illustrated for each configuration listed below.

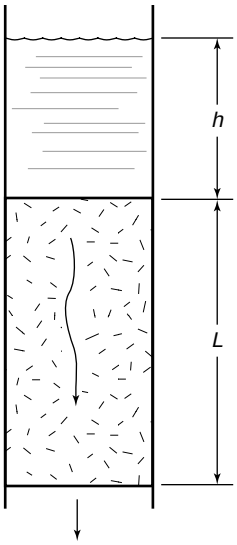


Figure 6-6—Downward Flow of Liquid with Driving Head, *h*

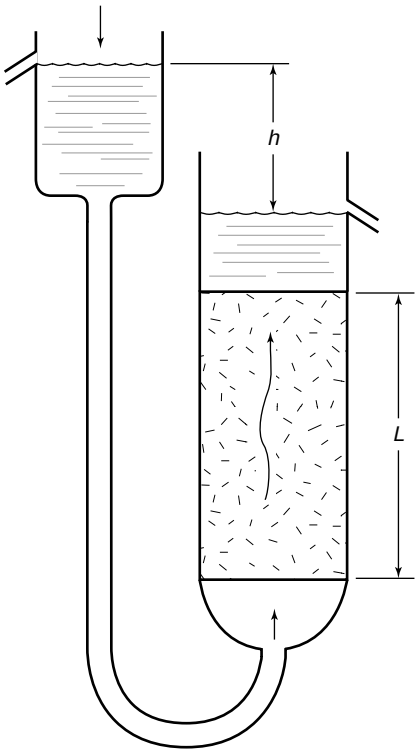


Figure 6-7—Upward Flow of Liquid with Driving Head, *h*

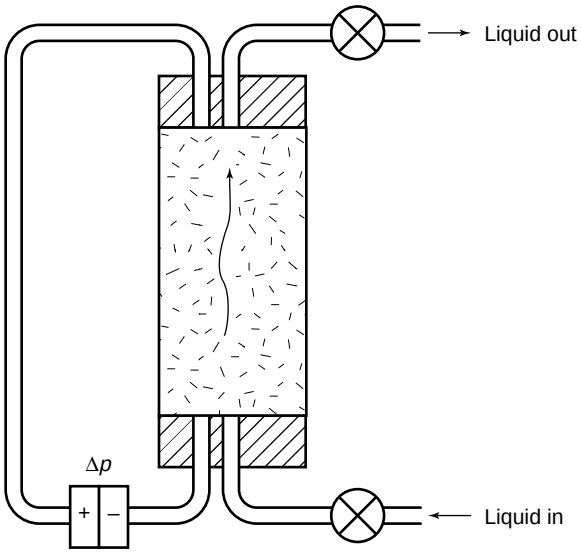
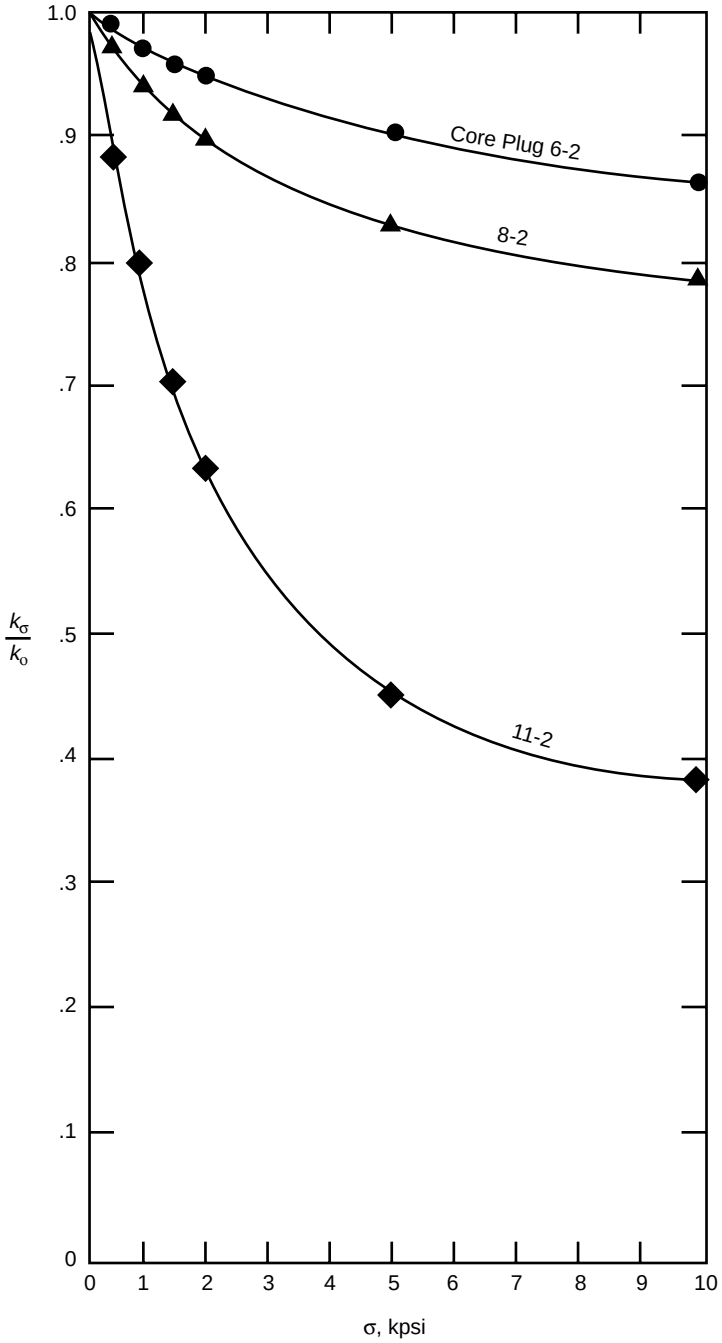


Figure 6-8—Upward Flow of Liquid with Imposed Pressure Difference, Δp



Note: Data from Jones[18].

Figure 6-9—Permeability Reduction with Increasing Net Hydrostatic Stress

6.3.1 Gases

6.3.1.1 Axial, Steady-State Flow of Gases

A schematic arrangement for axial flow of gases is shown in Figure 6-10. A cleaned and dried cylindrical core plug or whole core sample of length L and diameter D is mounted in a sample holder. The holder contains a flexible sleeve for purposes of making a gas-tight seal on the cylindrical walls of the sample, and for applying radial confining stresses. Axial stress can be transmitted to the sample by applying force to one or both end plugs, by mechanical, pneumatic, or hydraulic means. If the magnitudes of the radial and axial stresses are equal, the sample is said to be *isostatically* or *hydrostatically* stressed. If the magnitudes are unequal, the sample is biaxialy stressed. When gases at relatively low pressures (up to a few hundred psig) are used, gravity effects are negligible, and the sample holder may be oriented either horizontally or vertically.

The two end plugs are provided with an axial port for transporting gas to and from the sample. Each should have radial and circular grooves or other means for distributing gas to its entire injection face, and for collecting gas from all parts of its outflow face. Each end plug, preferably, also contains a second port for measuring upstream and downstream pressures, P_1 and P_2, respectively; the upstream pressure and differential pressure, Δp, as illustrated in Figure 6-10; or the differential and downstream pressures. However, lines for connecting the pressure-measuring devices can be teed into the flow lines near the axial ports, provided that the latter are sufficiently large not to cause significant pressure drops between each tee and the corresponding end face of the sample. It is essential that the branch of each tee (not the run) is connected to the pressure transducer. Otherwise, dynamic pressure effects may influence the pressure measurements.

Note that Δp, as used throughout this document is always a positive number, therefore is equal to $P_1–P_2$ or to $p_1–p_2$. As indicated previously, an upper case P denotes an absolute pressure, and a lower case p, a gauge or differential pressure.

The outflow line can be vented directly to atmospheric pressure (when the flow meter is upstream of the sample), connected to a flow rate measuring device, or to a back pressure regulator for purposes of creating elevated mean pore pressures. In the latter case, the flow meter can be either upstream of the sample or downstream of the back pressure regulator.

The volumetric flow rate, q_r, can be measured at the upstream or downstream pressure, or some other pressure, which in all cases is denoted as P_r, an absolute pressure. The temperature at which the flow rate is measured is assumed to be the same as the flowing gas temperature. Alternatively, the group $(q_r\ P_r)/(z_r\ T_r)$, which is proportional to mass flow rate, can be determined by a mass flow meter. See 6.6.2 for calibration of mass flow meters and application of results to the calculation of permeability.

6.3.1.1.1 Apparatus

6.3.1.1.1.1 Low Confining Stress

Figure 6-11 shows a simplified flow diagram for low pressure measurements. Figure 6-12 shows a low pressure Hassler-type core holder, in which a 400 psi radial confining stress and axial stress of unknown magnitude is typically applied to

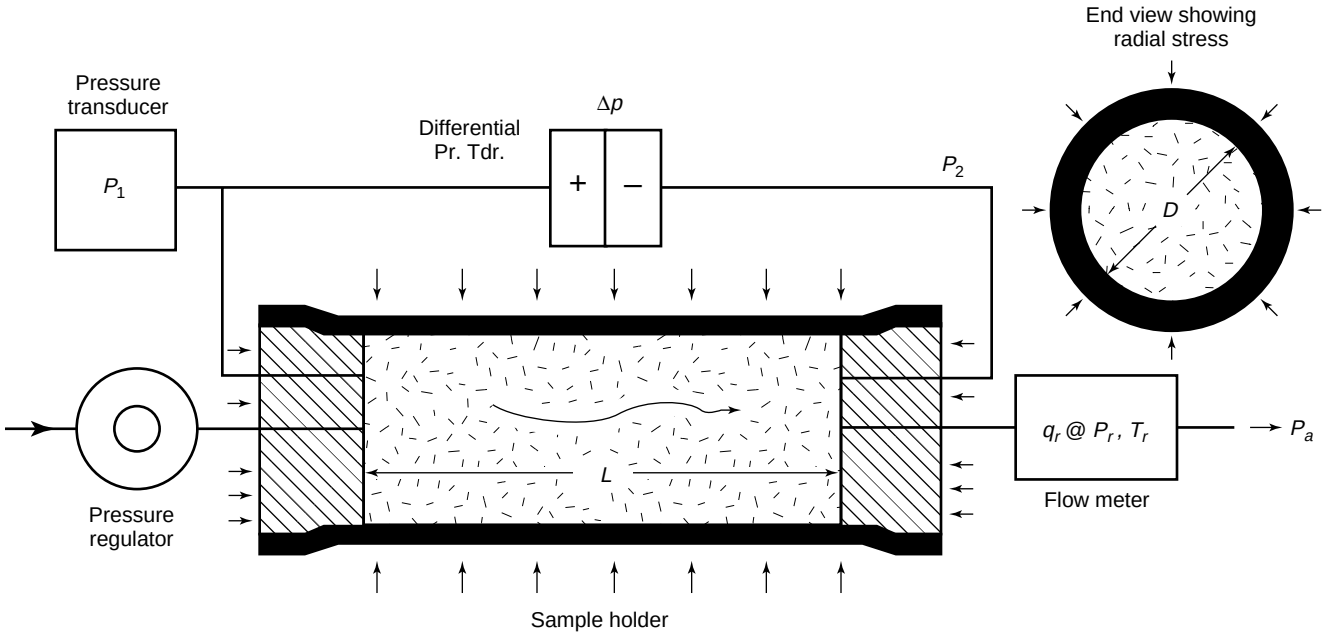


Figure 6-10—Schematic of Permeability Apparatus for Axial Flow of Gas

either a 1.0- or 1.5-inch (25- or 38-millimeter) diameter, 0.75- to 3-inch (19- to 76-millimeter) long core plug. These dimensions and stresses have become virtually standard in conventional, low stress core analysis. Radial stress is transmitted to the plug when pneumatic or hydraulic pressure is applied to the annular space between the rubber sleeve and core holder. Axial stress is typically transmitted from a screw that constrains the lower end plug. Normally, this stress is not measured, and is somewhat operator dependent.

Fancher-type core holders (see page 35 of API RP 40, 1960) are not recommended due to operator-dependent and non-reproducible sealing and confining stresses produced.

6.3.1.1.1.2 Elevated Confining Stress

A core holder for applying hydrostatic stresses, typically to a maximum of 10,000 psi, is illustrated in Figure 6-13. For safety, minimization of gas diffusion through the sleeve, and prevention of corrosion, hydraulic oil, or a heavy mineral oil is strongly recommended for stress generation. Axial stress in this core holder is reduced by the cross-sectional area of the tube connecting the internal end plug to the outer pressure vessel. For $^1/_8$-inch O.D. tubing, the reduction is not serious, but with larger diameters, the axial stress can be significantly less than the radial stress generated. For room temperature operation, 40 to 70 durometer (Shore A hardness), nitrile (Buna-N) rubber is recommended in sleeve thicknesses of 0.125 to 0.25 inch. The lower durometer and thickness can be used for lower stresses. Cold flow occurs with increasing stress and temperature. At stresses above about 6,500 psi, durometers higher than 70 are desirable for extended sleeve life.

Figure 6-14 illustrates a high pressure core holder that can be used to apply either hydrostatic or biaxial loading to core plugs. Axial stress is generated by a stress intensifier. Either pneumatic or hydraulic pressure bears on a piston of larger diameter than that of the end plug. The resulting force is transmitted by the lower end plug to the sample. The pressure required is inversely proportional to the ratio of areas. For example, if the larger piston has an area 10 times that of the end plug, a pressure of 600 psig applied to the large piston would transmit a stress of 6,000 psi to the core plug. If the axial stress is equal to the hydraulic pressure applied to the rubber sleeve, then the core plug is hydrostatically stressed. Otherwise, it is biaxialy stressed. In addition to its capability for providing unequal stresses, the main advantage of this type of core holder is that no disassembly is required for changing samples. Confining pressure is relieved (and often a vacuum is applied to expand the sleeve), the lower end plug is withdrawn, the previous sample is removed, and the new sample is inserted.

Strict safety precautions must be observed in both the design and operation of high pressure equipment. All parts that contain high pressures must suffer no permanent deformation at pressures at least equal to 1.5 times the maximum working pressure, or otherwise, must conform to local, national, or international codes that apply for high pressure vessels.

6.3.1.1.2 Calculations

Gas permeability, k_g, is calculated from Equation 19 (see 6.2.1.3). If multiple measurements are made at different mean pore pressures, k_∞ and b are obtained from a Klinkenberg plot

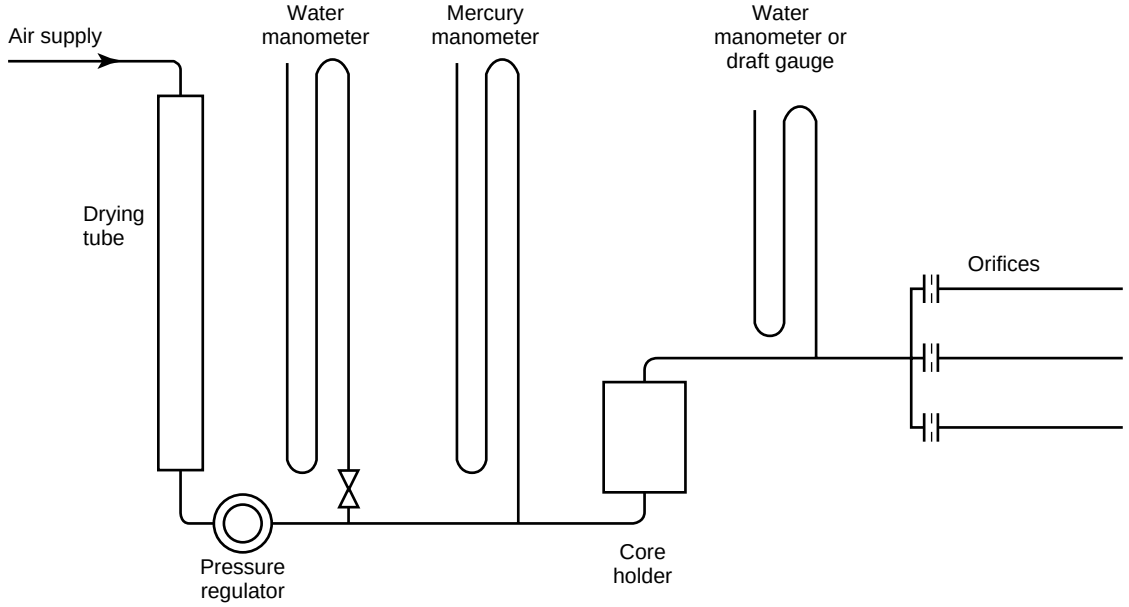


Figure 6-11—Simplified Flow Diagram for Low Pressure, Axial Gas Flow Permeability Measurements

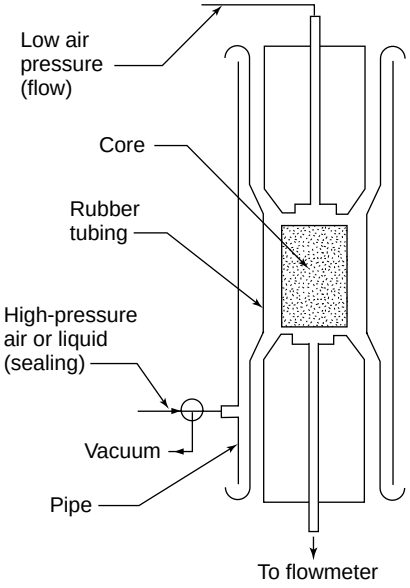


Figure 6-12—Low-Pressure Hassler-Type Core Holder

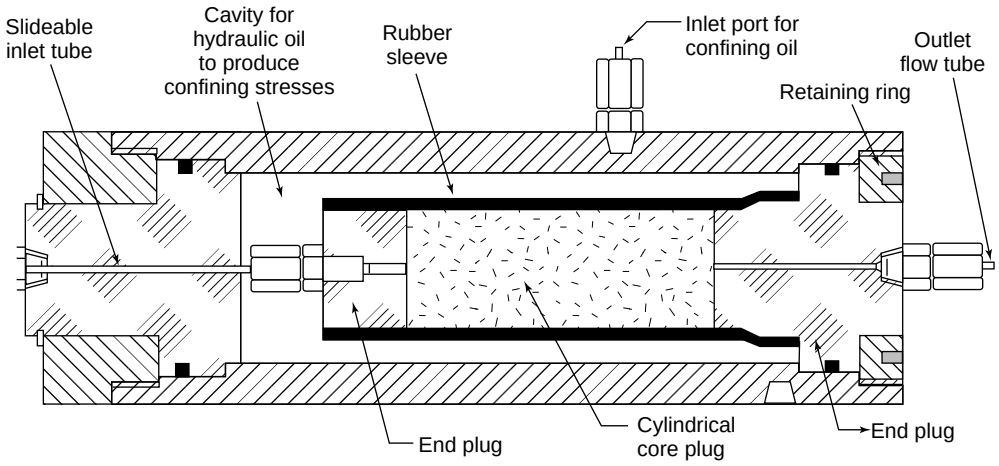


Figure 6-13—High-Pressure Core Holder for Hydrostatic Stresses

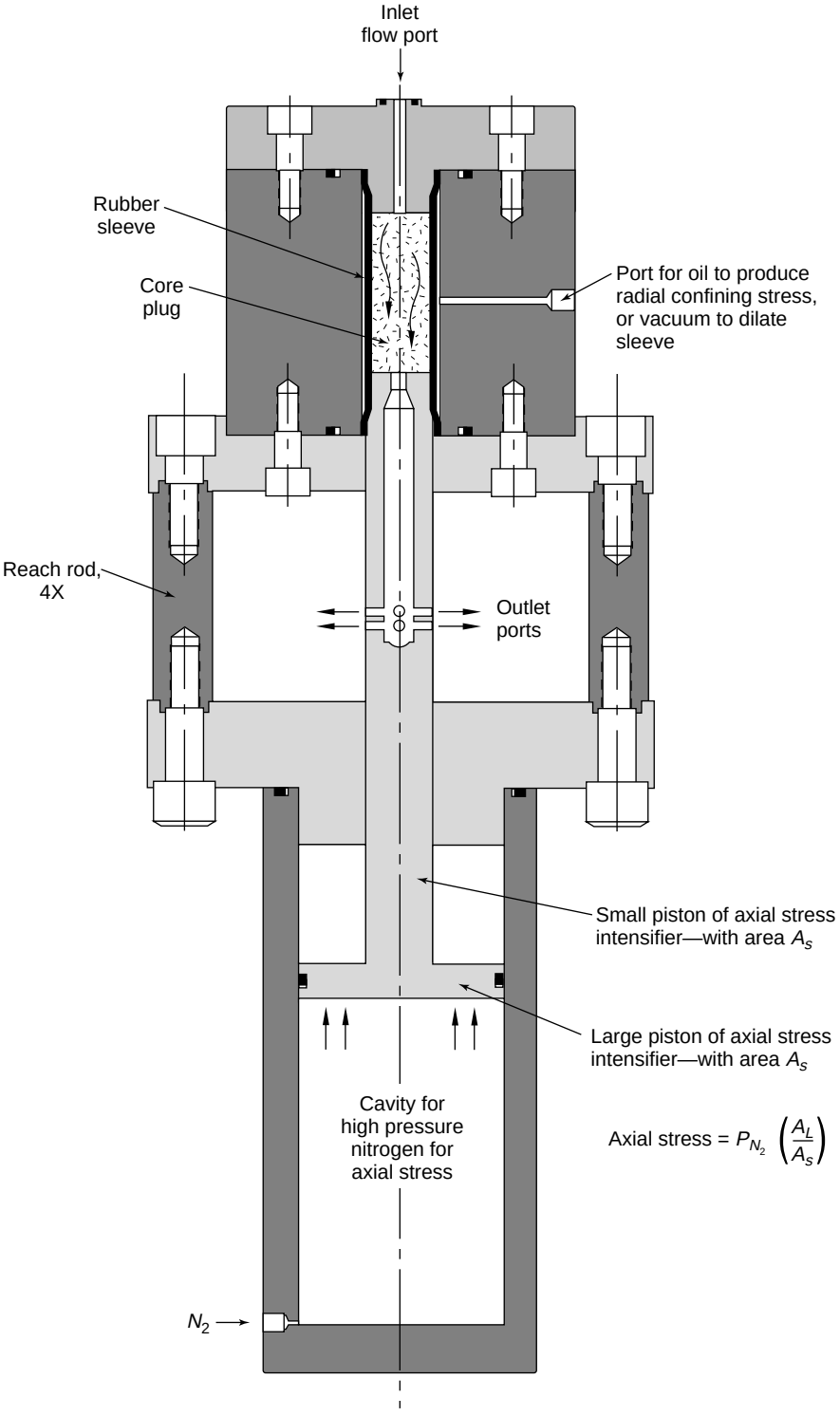


Figure 6-14—High-Pressure Core Holder For Hydrostatic or Biaxial Loading

(see Figure 6-5), or linear regression as described earlier. If b is found from a correlation, then k_∞ is calculated from Equation 18. These statements also apply to all other gas flow configurations listed below. The geometric factor for axial flow in Equations 18 and 19 is:

$$G_f = \frac{\pi D^2}{4L} \tag{27}$$

Traditionally, when a plug or whole core is cut so that its axis is parallel to bedding planes or permeability strike, the measured permeability is called its "horizontal" permeability, k_H. Similarly, a "vertical" permeability, k_V, is obtained when the sample's axis is perpendicular to bedding planes.

6.3.1.1.3 Advantages

Advantages of this method include:

a. The experimental method is simple and has been an industry standard for many years. Consequently, there is a large historic data base for direct comparison.
b. Gas does not react with rock, and samples are clean at the conclusion of measurements.
c. The technique lends itself to a wide range of equipment. With a low-pressure Hassler holder, manometers, orifice-type volumetric flow devices, and manual data acquisition, capital costs are low. However, an upgraded apparatus that includes a high pressure hydrostatic or biaxial core holder and automated control and data acquisition systems is strongly recommended.
d. Flow meters and pressure transducers can easily be exchanged to extend permeability range.

6.3.1.1.4 Precautions and Limitations

Precautions and limitations of this method include:

a. The lower practical permeability limit with this technique is about 0.1 millidarcy. In low permeability rocks the time required to achieve steady state becomes long, and flow measurements can become rather inaccurate.
b. The primary disadvantage of non-slip-corrected, low stress measurements (typically, 400 psi radial confining stress, and unknown axial stress) is that they over estimate in situ reservoir permeability, especially in tight samples. The Klinkenberg slip correction can be made from measurements at several mean pore pressures, but routine measurements are typically made at only one pressure, and this pressure is seldom reported. If this pressure is not reported, the permeability is difficult to normalize, and is incompletely specified. The gas used and the mean pore pressure must be reported. If a gas slippage correction is made from a correlation, this fact must be reported.
c. Attainment of Stokes flow, especially with high permeability samples, can be difficult to ascertain with single point measurements. Measurements should be checked per section 6.2.1.3 to make sure that the maximum allowable $\Delta p/L$ has not been exceeded.
d. It is critical to cut perfectly square ends on core plugs, especially with high confining stresses. A high point will receive a disproportionately high stress, which may crush this portion of the plug. This, of course, applies to all techniques involving cylindrical core plugs.

6.3.1.2 Steady-State Probe Permeability Measurements With Gases

When gas flows from the end of a small-diameter tube (or "probe") that is sealed against the surface of a slabbed or unslabbed whole-core sample, or a permeable rock outcrop, the flow pattern is somewhat similar to that of hemispherical flow (see Figure 6-15). This configuration is widely used in making inexpensive, non-destructive, zero-stress gas permeability measurements within small radii of influence. If the cores are not cleaned and dried, a measurement yields an effective permeability at some unknown oil and water saturation. Such measurements can be made at close spacing to determine permeability variation in a heterogeneous formation. Instruments for making these measurements are generically called "probe permeameters," or "mini-permeameters."

True hemispherical flow geometry would require: (a) a large, homogeneous, isotropic sample with a plane upper surface, (b) a hemispherical cavity under this surface, immediately under the probe, with a radius, r_i, equal to the inside radius of the probe seal, (c) a very large (infinite) extent of the sample in all directions under the plane of contact, and (d) a seal everywhere on this plane except over the hemispherical cavity. Because these conditions are totally impractical, Goggin, et al.[19] performed numerical calculations to produce dimensionless geometrical flow factors that account for the lack of a hemispherical cavity on the surface of a small, finite sample, and a probe seal of small, finite interior and exterior radii.

These dimensionless factors are displayed in Figure 6-16 as a function of the ratio of the outer radius, r_o, of the probe seal to its inner radius, r_i. For true, semi-infinite, hemispherical flow, G_0 would equal 2π. The relationship shown in Figure 6-16 is intended for samples with a plane upper surface and sufficient lateral extent and depth that exterior boundaries have no influence on permeability measurements. For practical purposes, the factors shown can be used with little error on samples that have a depth at least equal to four times the interior radius of the probe seal, and where the nearest lateral boundary is at least $4r_i$ from the axis of the probe. Also, little error is introduced if the probe is placed on a cylindrical surface of a sample, provided that a positive, gas-tight seal is made, the radius of the sample is at least $12r_i$, and the above sample boundary restrictions are observed. See Goggin, et al.[19] for dimensionless geometric factors that relate to nearer boundaries.

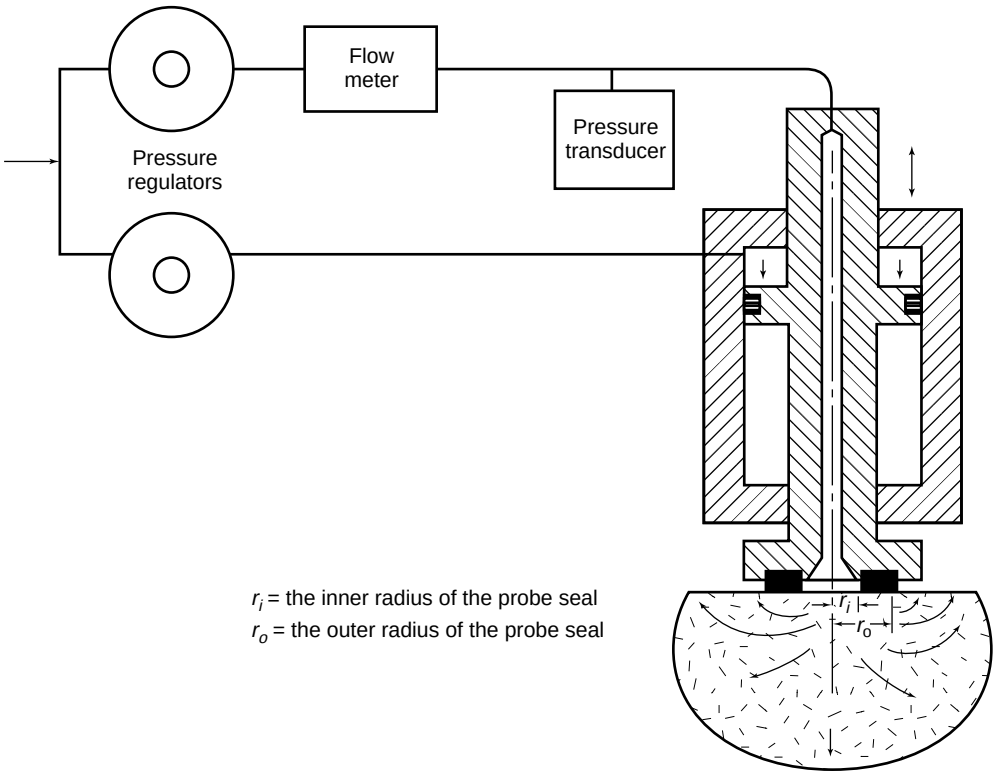
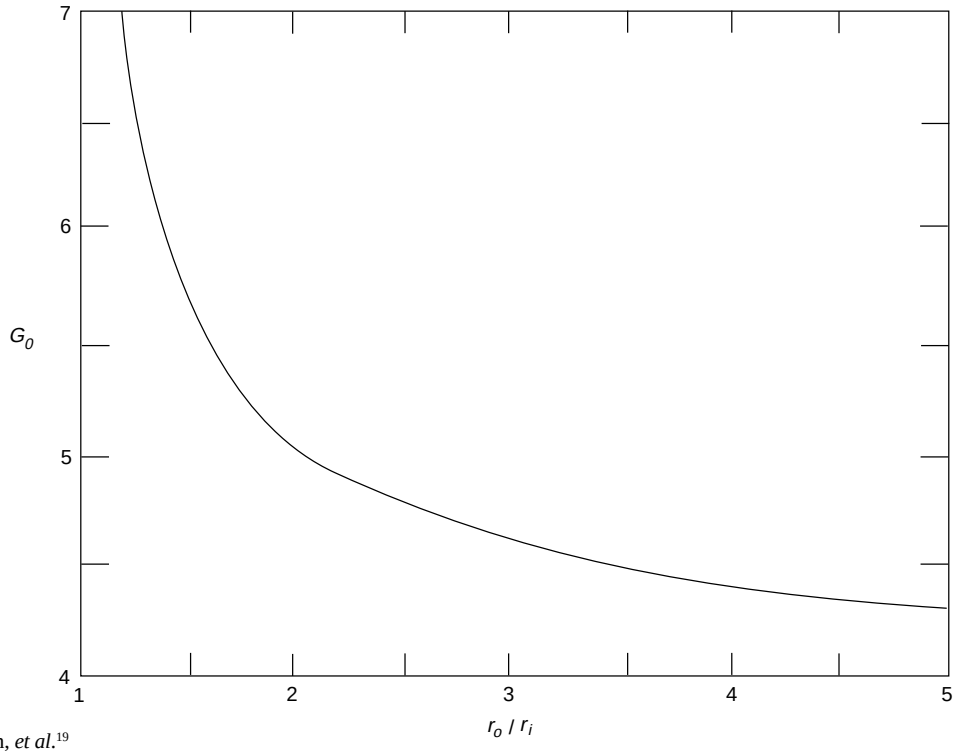


Figure 6-15—Schematic of Steady-State Probe Permeameter



Note: From Goggin, *et al.*[19]

Figure 6-16—Dimensionless Geometric Factor for Measurements with Probe Permeameter

6.3.1.2.1 Apparatus

For gas permeability measurements (see Figure 6-15), the probe must be provided with a soft rubber seal that has dimensionally stable (or reproducible) r_i and r_o when pressed against the sample, and means for delivering a constant, reproducible sealing force to it. This sealing force must be adequate to prevent gas from escaping anywhere on the contact surface between the seal and the rock. The inner radius of the seal is typically 2 to 3 millimeters, and the ratio of its outer to inner radii is typically 1.5 to 2.5. The smaller radii and ratios yield more resolution with regard to small scale heterogeneity, and larger values are used to obtain more permeability averaging. These dimensions must also be scaled to the coarseness of the rock texture.

The probe is positioned above a desired point on a sample, which must be related to depth and position relative to the core's axis or some other reference point. The probe is lowered to the rock's surface, and sealed against it. Gas at pressure P_1 is delivered from the probe to the sample. It exits from the sample at atmospheric pressure. Provision must be made for regulating pressure in the probe, and for measuring it and the volumetric gas flow rate at this pressure. Flow meter range is selected on the basis of the permeability being investigated. Pressure and flow rate are recorded when both become constant.

6.3.1.2.2 Calculations

From these measurements, plus the seal dimensions, atmospheric pressure, and gas viscosity, k_g can be calculated from Equation 19, wherein, if p_1 is the measured upstream gauge pressure:

$$P_1 = p_1 + P_a \quad ,$$

$$P_2 = P_a \quad ,$$

$$P_r = P_1 \quad ,$$

and

$$G_f = G_0 r_i \tag{28}$$

The permeability obtained is most heavily weighted (in non-homogeneous samples) within a radius equal to the inside seal radius, and progressively less with increasing radii. Because of the small volume of rock investigated, permeability on a macro scale must be obtained by appropriate averaging techniques.

6.3.1.2.3 Advantages

Advantages of this method include:

a. The method is nondestructive in the sense that there is no need to cut core plugs. It is rapid and low cost, thereby permitting high-density sampling.

b. The probe investigates only a small volume of rock. The measurement is well suited for investigation of spatial permeability variation in cores containing thin laminations and small scale heterogeneity. Directional permeability variation around the circumference of a whole core can be measured.

c. The device can be engineered for portable well site service, and for use on outcrops, provided that an adequate seal can be achieved.

d. With multiple flow meters a permeability range of about 1 to 10,000 millidarcys can be measured.

6.3.1.2.4 Precautions and Limitations

Precautions and limitations of this method include:

a. Minimum flow path lengths in a sample (from r_i to r_o) range from 0.2 to 0.4 cm for typical probe tip seals. Most of the total pressure drop occurs within this distance, which is an order of magnitude shorter than the path length for axial flow through most core plugs. The injection pressure, p_1, must be reduced correspondingly to avoid significant inertial flow resistance. Even though Goggin, et al.[19] proposed a "high velocity flow correction" using a correlation to obtain β, this correction can only be approximated with single-rate measurements, because β varies by plus or minus at least an order of magnitude at a given permeability for most permeability ranges. However, this correction is smaller the lower the injection pressure.

b. Nearly zero stress is applied to the rock during a measurement. Therefore, permeabilities tend to be very optimistic, especially in tight samples. If Klinkenberg corrections are not made (from a correlation), permeability is further overestimated, especially in low permeability rock.

c. Unless the tip seal is laterally confined, and essentially flat on the bottom (in the unstressed condition) its geometric factor can be very sensitive to the sealing stress applied. This stress, therefore, must be highly reproducible.

d. Although the technique can be used on whole core segments, slabbing is strongly recommended to remove mud solids invasion and other surface contamination, and to provide a flat, smooth surface for probe tip sealing, which otherwise is hard to guarantee.

e. If the surface of the core is not dry, measured permeability may be erroneously low due to severe relative permeability effects. Permeability may increase with time, then stabilize as mobile liquids are evaporated or blown away from the probe tip.

f. Probe tip seals should be inspected regularly for abrasion, deterioration, and imbedded sand grains, and replaced as needed. Periodic leak-testing on a smooth, flat impermeable surface is useful for detection of flaws or inclusions.

g. Due to the small volume of rock investigated by this technique, permeabilities on a macro scale must be obtained by appropriate averaging techniques.

6.3.1.3 Transverse, Steady-State Flow of Gases

This technique is used primarily to measure directional, "horizontal" permeability in (but not limited to) whole core samples. Gas flows from an inlet screen, covering a subtended angle θ and the entire length, L, of a whole-core sample (or core plug), across the sample, and into a similar outlet screen placed diametrically across from the inlet screen (see Figure 6-17). The flow pattern is complex, and the area normal to the streamlines for gas flow is variable throughout the flow path. Collins[20], using a conformal mapping transformation, computed a dimensionless geometrical factor, G_θ. It is shown in Figure 6-18, and is a function of the angle subtended by the screens. Permeability is normally measured in two directions—one giving the maximum value (usually along the direction of principal fracturing or permeability strike), and the other at 90 degrees to the maximum.

6.3.1.3.1 Apparatus and Calculations

The flow system and measurements required for calculation of transverse flow permeabilities are similar to those for steady-state axial flow. Figure 6-19 shows a Hassler-type core holder for transverse permeability measurements. Gas is allowed to enter and leave the sample through diametrically opposed openings through the sleeve. These connect to two mesh screens that cover the entire length of the sample and a known subtended angle on its circumference. If the subtended angle of each screen is 90 degrees, then the dimensionless geometrical factor, G_θ, is equal to 1.0. The seal materials must not penetrate the mesh and restrict flow. Both ends of the sample are sealed with soft rubber disks.

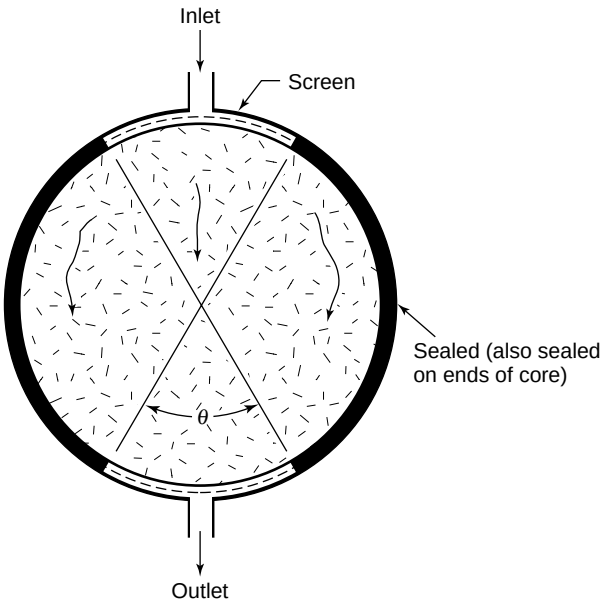
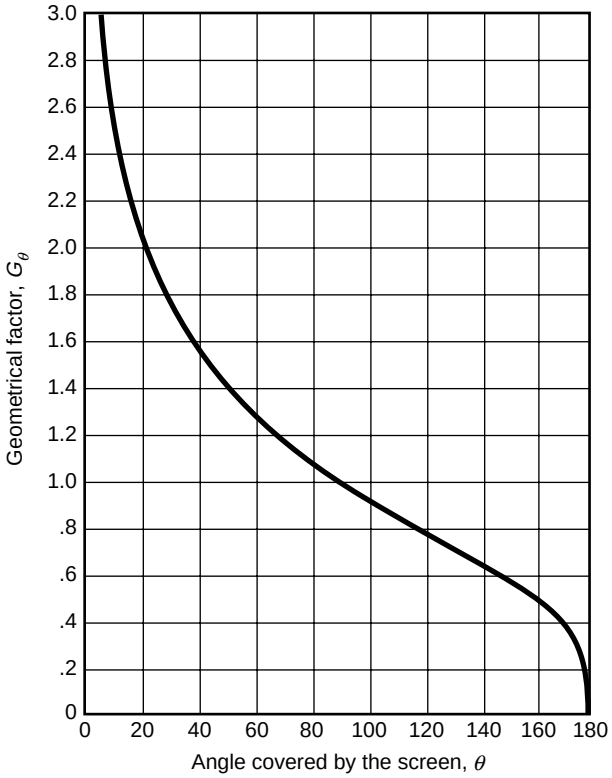


Figure 6-17—Transverse Flow Configuration for Gases or Liquids



Note: From Collins[20].

Figure 6-18—Dimensionless Geometric Factor for Transverse Flow

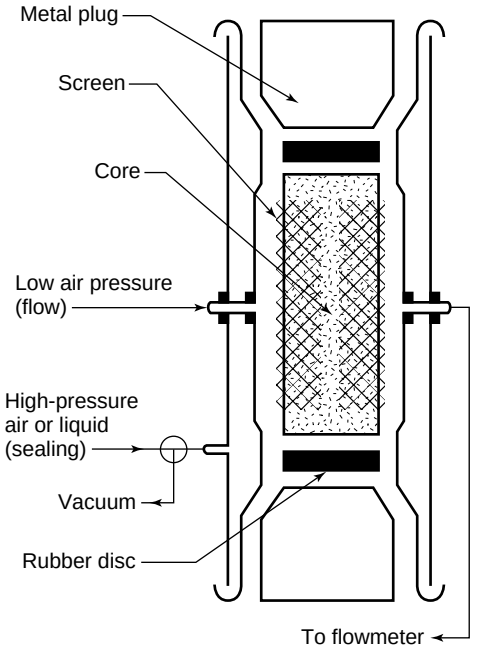


Figure 6-19—Hassler-Type Core Holder for Transverse Permeability Measurements

The dimensional geometric factor to be used in Equations 18 or 19 for transverse flow is:

$$G_f = \frac{L}{G_\theta} \tag{29}$$

A whole core sample can be placed in a large core holder for longitudinal "vertical" permeability measurements, before or after the transverse measurements. Calculations are those for axial flow (see 6.3.1.1.2).

6.3.1.3.2 Advantages

Advantages of this method include:

a. Measurements on whole core samples involve large volumes of rock, thereby permitting considerable permeability averaging. This minimizes the effect of small scale heterogeneity.

b. On oriented whole core samples, orthogonal measurements can be made to determine directional permeability.

6.3.1.3.3 Precautions and Limitations

Precautions and limitations of this method include:

a. A whole core sample is seldom cut precisely normal or parallel to the rock's bedding planes. Therefore conventionally defined "vertical" and "horizontal" permeabilities may not be obtained exactly with longitudinal and transverse measurements, respectively, on these samples.

b. Larger samples take considerably longer to clean, dry, and reach steady state. Experiments therefore take longer and are more expensive than those on core plugs.

c. The same precautions relating to gas slippage and inertial resistance, given in 6.3.1.1.4 for axial flow, should be observed.

6.3.1.4 Radial, Steady-State Flow of Gases

Figure 6-20 shows the flow configuration for steady-state radial flow. The upper and lower faces of a clean, dry cylindrical sample of length L and exterior radius r_e, into which an axial bore of radius r_w is cut, are sealed everywhere except over the central bore. Gas at pressure P_1 flows from the unsealed cylindrical surface radially to the central bore, and exits at pressure P_2. Otherwise, the flow system and measurements are the same as for axial flow. In principle, the flow direction can be reversed, so that gas is injected into the central bore at pressure P_1, and exits through the sample's exterior cylindrical surface at pressure P_2. However, injection into the well bore is generally not recommended because its smaller flow area is more prone to damage from trace amounts of particulates in the injected fluids.

6.3.1.4.1 Apparatus and Procedure

The full diameter radial permeameter, shown in Figure 21, consists of three parts: the cell, which is sufficiently large to maintain a uniform inlet pressure; a piston to apply the sealing force; and the floating plate assembly that consists of a lower fixed plate, a pivot ball, three springs 120 degrees apart, and the upper floating plate.

The core is placed on a 1-inch thick solid rubber gasket that is attached to the lower floating plate. The core is then raised against the closed lid, the center hole of the core matching that of the upper gasket. As the piston pressure increases, the lower floating plate automatically adjusts if the ends are not parallel. To check for an air leak between the ends of the core and the rubber gaskets, the piston pressure is increased. A decrease in the flow rate indicates that a leak had existed. This test is repeated until no change in flow rate is noted.

6.3.1.4.2 Calculations

Flow rates and pressure drops are measured and used in the same manner as for axial flow measurements. Steady state is reached when both become invariant with time. Permeability, k_g, is calculated from Equation 19, wherein the geometrical factor for radial flow is:

$$G_f = \frac{2\pi L}{\ln[r_e / r_w]} \tag{30}$$

Average permeability of non-homogeneous samples is most heavily weighted by the permeability at the inner wellbore radius, r_w (where the pressure gradient is greatest), and progressively less at larger radii.

6.3.1.4.3 Advantages

The measured permeability is averaged throughout the length of the sample and in all radial directions.

6.3.1.4.4 Precautions and Limitations

Precautions and limitations of this method include:

a. Permeability is critically dependent upon the internal wellbore radius. Small errors in its value, especially with small diameter holes, will cause large permeability errors. Similarly, induced fractures or surface damage near this hole will greatly affect apparent permeability.

b. Sample preparation for this measurement is not easy. Therefore the test tends to be somewhat more costly than alternative configurations.

c. Axial stresses are not balanced by radial stresses. The method is not easily adapted to application of elevated stresses for measurement of realistic in situ reservoir permeability.

d. A large piece of core material is altered by the central bore hole required for this technique, and the sample cannot usually be used for other tests.

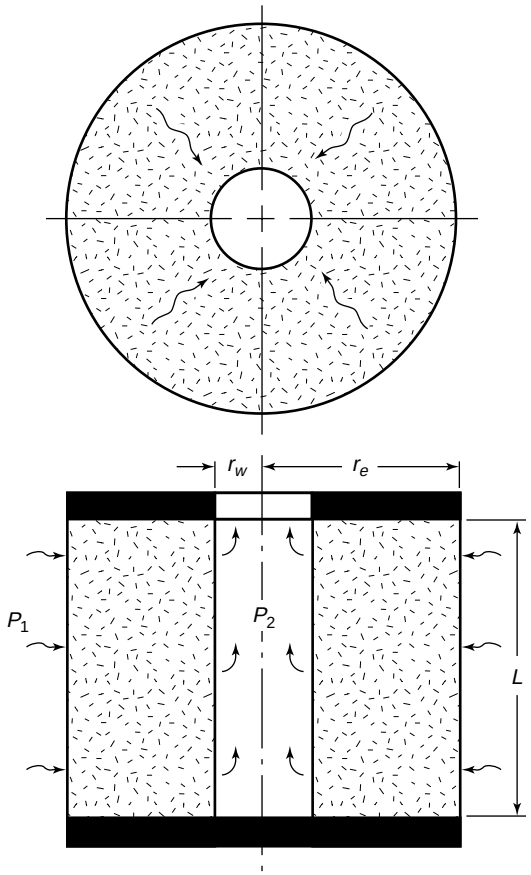


Figure 6-20—Schematic for Inward Radial Flow

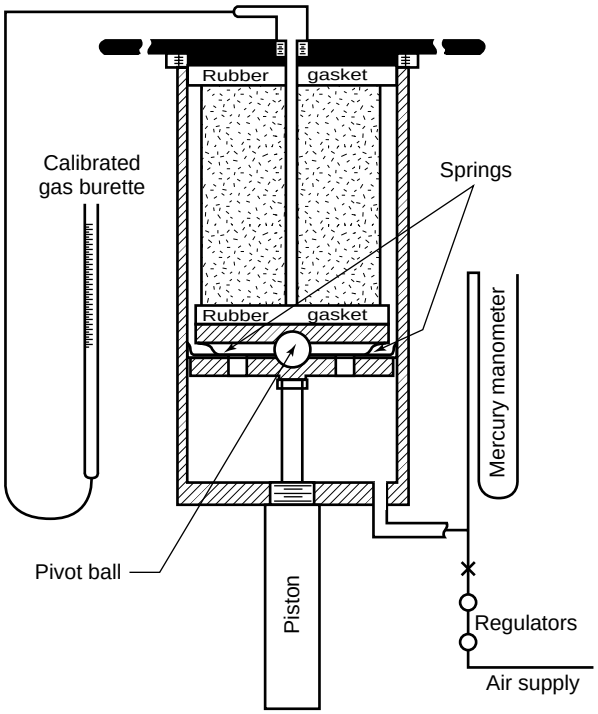


Figure 6-21—Full-Diameter Radial Flow Permeameter

6.3.2 Liquids

Steady-state liquid permeability apparatus have many features in common with those for gas. Differences between liquids and gases that may require equipment modifications are: liquid viscosity and density are considerably greater and compressibility is much smaller than the corresponding gas properties. Furthermore, liquids (especially aqueous solutions containing salts) can be corrosive, support microbial activity, and react with clay and mineral constituents of the rock.

Permeability to liquids is often observed to be lower than slip-corrected gas permeability. This can be due in part to liquid-clay interaction (especially with aqueous solutions), fines migration with high liquid flow rates, or incomplete liquid saturation.

6.3.2.1 Steady-State Liquid Permeability Measurements with an Imposed Δp

6.3.2.1.1 Apparatus

This type of permeability apparatus does not depend upon liquid heads for delivery of fluid. It comprises three separate units: sample holder, pressure measurement system, and fluid delivery system. Except for corrosion resistance and compatibility of the rubber sleeve with a particular liquid, the sample holder, delivery lines, and pressure measuring equipment for liquid permeability measurements are not different from systems for gases. The higher liquid density requires that attention be paid to liquid heads. These normally cancel out with horizontal flow, provided that both ports of the differential pressure transducer used are at the same elevation, and that all pressure-measuring lines are completely filled with the same fluid as the flowing fluid, and are at the same temperature. In this case, when flow through the sample is terminated, the Δp reading should go to zero. Under these circumstances Equation 23 (see 6.2.2.1.1), which applies to horizontal flow, also becomes valid for upward or downward vertical flow (see comments following Equation 26 in 6.2.2.1.2).

Because of the 50-fold (or more) higher viscosity of most liquids compared to gases, liquid flow rates are correspondingly lower at the same pressure gradient. The fluid delivery system is normally either a primary constant flow rate source or a constant pressure source that can be regulated to result in a constant flow rate. Single-stroke, positive displacement pumps offer the most accurate means of delivering a liquid at a controlled constant rate. The use of such pumps precludes the need to collect produced liquids from a core to determine

flow rate. However, the volume contained in a single-stroke pump may be insufficient to reach steady state. To completely eliminate pressure upsets and flow rate discontinuities, two such pumps and a sophisticated control system are recommended for a "once-through" flow system, and three are desirable for a recirculating flow system.

Slow attainment of steady state, provided that the sample's permeability is not changing due to progressive plugging, clay or mineral interaction, etc., is due to high compressive storage in the system. Compressive storage is the product of the volume and effective compressibility of each component in the system. Effective compressibility is the sum of fluid and vessel compressibilities. Fluid compressibility can be reduced markedly (if any gas is present) by applying a high back pressure to the system, thereby dissolving gas bubbles. Care should be taken to remove trapped gas from the system, and to de-gas liquids by applying a vacuum. Vessel compressibility is reduced by making vessels more rigid. System volumes should be minimized wherever possible. Use of transfer vessels (where, for example, oil from a pump displaces brine that is injected into a core plug) at least doubles the volume, compared to displacing brine directly from the pump. However, corrosion considerations may preclude direct pumping. Whenever compressive storage is nearly zero and permeability remains constant, achievement of steady state is virtually instantaneous.

When using a chromatography-type pump, the operator should ensure that its delivery is tested over the entire range to be employed. To calculate permeability, flow rates should be measured independently. This can be accomplished by collecting and weighing produced liquid in a weighing vessel on an electronic balance at frequent, timed intervals. Volumetric flow rate is calculated by dividing the rate of mass accumulation by the liquid's density.

Constant pressure systems utilize either a gas or liquid source to displace de-gassed liquid from a vessel and through the sample. The liquid to be displaced is contained in a piston accumulator or collapsible rubber bladder. The displacing fluid passes through a pressure regulator. As for the case of chromatography type pumps, flow rate must be determined independently.

6.3.2.1.2 Procedure and Calculations

The liquid to be used should be well-filtered through a fine filter shortly before use. A 0.2 micron filter will remove bacteria as well as solid particles. A fully saturated test sample should be carefully loaded into an appropriate sample holder, ensuring that no air is trapped in the process. Confining stresses are then applied. In the absence of any other permeability data, a low Δp or flow rate should be applied initially to allow the operator to make an initial approximation of permeability, and thereby set conditions of flow rate and pressure drop, and select appropriate equipment for these measurements. See A.6.8.1 to determine the maximum $\Delta p/L$ to avoid significant inertial resistance. However, this is generally not a serious problem with liquids. Care should be taken to avoid the mobilization of rock fines. The effluent should be collected and observed for contamination. Confining stresses should be adjusted to obtain the desired net stress.

Close temperature control must be maintained to ensure constant viscosity, and avoid volumetric flow rate changes with liquid expansion or contraction. This is particularly critical when large vessels are involved, accompanied by low displacement rates from a positive displacement pump, with low permeability plugs. Huge flow rate variations can occur in this situation with small changes in temperature over short time intervals. The critical factor is the thermally induced volumetric expansion rate, Δq:

$$\Delta q = \beta_T V\left(\frac{dT}{dt}\right)$$

relative to the pump's displacement rate.

Where:

β_T = coefficient of volumetric thermal expansion of liquid, $°C^{-1}$ or $°F^{-1}$.

V = volume of liquid being displaced.

$\frac{dT}{dt}$ = time rate of temperature change.

To illustrate, suppose that a positive displacement pump contains 1,000 cm^3 of decane that is being injected into a low permeability plug at a nominal pump displacement rate of 1.000 cm^3/hr., and that the temperature of the decane is increasing at a rate of 0.02°F/minute, due to changes in the ambient temperature. The coefficient of thermal expansion of decane is about 5.5×10^{-4} volumes per volume per degree F. Therefore the rate of thermal expansion of decane is $5.5 \times 10^{-4} \times 1000 \times 0.02 = 0.011$ cm^3/min. With the pump's displacement rate of 0.0166 cm^3/min, this causes an error in flow rate of 66 percent of the nominal rate! If the displacement rate were increased by a factor of 10, the error would be reduced to 6.6 percent. Additionally, if the pump's volume were reduced to 10 cm^3, the error would be only 0.066 percent. Another way to reduce volumetric expansion is to increase the thermal mass of the pump, and improve its insulation.

With a constant-pressure delivery system (in which flow rate must be determined independently), thermal expansion of liquid in its displacement vessel is not a problem, provided that the upstream pressure regulator is self-relieving. In this case pressure will remain constant in spite of thermal expansion.

After both flow rate and differential pressure have stabilized they should be read and recorded, then the flow terminated. The transducer lines must be completely filled with the

test liquid, and both ports must be at the same elevation. After the differential pressure stabilizes with zero flow, it is read. If it is not zero, the reading should be subtracted from the stabilized, flowing Δp. Liquid permeability is calculated from Equation 23, using the appropriate dimensional geometric factor, G_f. These factors for axial flow, transverse flow, and radial flow are given by Equations 27, 29, and 30, respectively. With the above transducer line restrictions, Equation 23 applies to vertical, upward or downward axial flow as well as to horizontal flow for all three geometric configurations.

After the measurement has been completed and the sample is to be removed for further analysis, care should be taken to ensure that, as the sample dilates while confining stress is reduced, liquid is imbibed and the ingress of air is precluded.

6.3.2.1.3 Advantages

Advantages of this method include:

a. Pressure differentials for Stokes flow with liquids are higher and easier to measure than those with gases, especially for high permeability samples.
b. No gas slippage correction is required.
c. Carefully made liquid permeability measurements may be more representative of reservoir permeabilities.
d. Sample drying, a potentially damaging operation, is not required.
e. Liquid permeability measurements may be advantageous if additional analyses require the plug to be saturated with the same liquid.

6.3.2.1.4 Precautions and Limitations

Precautions and limitations of this method include:

a. Saturation and preparation of the rock sample, and preparation and handling of liquids is generally more difficult and time consuming than preparations for gas measurements.
b. Corrosion resistant, high pressure pumping and control equipment is expensive.
c. Care must be taken to avoid fluids that interact with rock constituents.
d. Steady state liquid measurements with low permeability plugs may be difficult to make accurately, and require long periods to reach steady state. Small volumes, short plug lengths, low compressive storages, and very tight temperature control are strongly recommended.

6.3.2.2 Steady-State Liquid Permeability Measurements Using Liquid Heads

Figures 6-6 and 6-7 (Section 6.2.2.1.2) show two configurations for axial flow measurements in which the liquid is delivered at a constant rate and pressure by means of constant liquid heads. The configuration of Figure 6-7 is particularly useful for accurate, low cost measurements with moderate to high permeability core plugs. No pump or pressure transducer is required. The adjustable-height inlet reservoir and fixed outlet reservoir are both provided with overflow drains. Liquid is allowed to flow into the inlet reservoir from another, higher-elevation supply vessel through a metering valve at a slightly higher rate than the rock sample will accept. Excess liquid flows out the drain vent and is collected for future recycle to the supply vessel. Liquid overflowing from the outlet reservoir drops into a weighing vessel on an electronic balance. Volumetric flow rate, q, is calculated by dividing the rate of mass accumulation (from weight and elapsed time measurements) by the density of the liquid, which must be determined. Permeability is calculated from Equation 25, where ρ is the density of the liquid at the measurement temperature, g is the local value of gravitational acceleration, and h is the elevation difference between the free surfaces of the two liquid heads. Because very little back pressure is exerted, extreme care must be taken to exclude air from all portions of the system, including the core plug.

For very high permeability samples (10 to 40 darcys), the configuration of Figure 6-7 is useful in that the overflow vent in the downstream vessel minimizes variation in the liquid head due to capillary retentive forces. These forces can be significant in the tip of small diameter exit nozzles or ports, creating a large relative variation in the Δp.

6.4 THEORY AND APPLICATION OF UNSTEADY-STATE PERMEABILITY DETERMINATIONS

The advent of high speed data-acquisition systems, accurate pressure transducers, and digital computers has made it not only feasible, but convenient to measure permeabilities under transient, or unsteady-state flow conditions. Transient measurements employ fixed-volume reservoirs for gas or liquid. These may be located either upstream of the sample—from which the gas or liquid flows into the sample being measured, or downstream—into which it flows from the sample, or in both places. When fluid flows from an upstream reservoir, its pressure in that reservoir declines with time. Similarly, when fluid flows into a downstream reservoir, pressure there builds with time. Instantaneous flow rates can be calculated from the volume of the reservoir and the instantaneous rate of pressure change, obviating the need for a flow-rate measuring device.

When fluid expands, it performs flow work at the expense of loss of internal energy. This is observed as a decrease in its temperature. Similarly, when fluid is compressed, work is done on it, and its temperature increases. Because instantaneous flow rates are calculated from rates of pressure change, it is mandatory to maintain isothermal conditions, or to measure instantaneous temperatures and use appropriate mathematical formulations. Equations presented herein assume isothermal conditions.

6.4.1 Transient Pressure Techniques For Gases

Fortunately, gas has low heat capacity, and work-related temperature changes can be virtually eliminated by fabricating reservoirs from material of high thermal conductivity and packing them with copper tubes parallel to the flow axis. Also, where high flow rates are anticipated (with high permeability samples), helium is the gas of choice. Its thermal diffusivity is much higher than that of air or nitrogen.

Other potential thermal problems can arise from changes in ambient temperature in long-duration measurements of low permeability samples, and from Joule-Thomson expansion as gas flows through a sample. The first is minimized with excellent thermal insulation, improved ambient temperature control, and by reducing measurement time. Joule-Thomson expansion is best handled by using helium, which increases in temperature (at ambient conditions) upon expansion, unlike all other gases except hydrogen. This warming tends to counteract work-related cooling.

Two main categories of transient pressure techniques have emerged for measuring permeability in the laboratory. One is called the "pulse-decay" method. It is characterized by using both upstream and downstream reservoirs, one or both of which are relatively small in volume. These and the sample are filled with gas to a fairly high pressure, 1,000 to 2,000 psig, which reduces gas slippage and compressibility. After pressure equilibrium is achieved throughout the system, pressure in the upstream reservoir is increased, typically by 2 to 3 percent of the initial pressure, causing a pressure pulse to flow through the sample. This technique is well suited for low permeability samples, 0.1 millidarcy to about 0.01 microdarcy. Small differential pressures and low permeabilities virtually eliminate inertial flow resistance. Only "late time" techniques will be discussed in this document. They yield overall permeabilities that are comparable to steady-state values. "Early time" transients provide information regarding heterogeneity in samples, which is beyond the intended scope of this document.

The other technique is called the "pressure falloff" method. It is characterized by upstream reservoir(s) only. The downstream end of the sample is vented to atmospheric pressure. The maximum upstream pressure used is fairly low, 10 to 250 psig (varying inversely with the permeability to be measured). A single transient pressure falloff produces data for 6 to 30 separate permeability calculations, each at a different flow velocity and mean pore pressure. Adequate variation of flow conditions during a single transient test makes possible the calculation of slip-corrected (Klinkenberg) permeability (k_∞), Klinkenberg slip factor (b), and inertial resistivity (β) of the porous medium. This technique, which has a useful permeability range of 0.001 to 30,000 millidarcys (through the use of multiple upstream gas reservoirs and pressure transducers), complements the pulse-decay method. For high permeability samples (>1,000 md), where b is small, it is difficult to determine b accurately when a sample is vented to atmospheric pressure. It is more reliably approximated from a correlation.

In addition to physical differences between flow systems, approaches to the derivation of flow equations are quite different for the pulse-decay and pressure-falloff techniques. In the former, Darcy's equation and the continuity equation (which is a statement of the conservation of mass) are solved simultaneously. In the pressure-falloff method, the steady-state solution of the slip-corrected Forchheimer equation is used as a starting point. This solution, which does not properly account for the increase in mass flux with distance along a sample's length at a particular instant (with non-steady-state flow), is differentiated. It is then inserted into the continuity equation, which is integrated to provide a correction (and improvement) to the final flow equation. This process is iteratively repeated until both the Forchheimer and continuity equations are satisfied.

The deviation between steady-state and pressure-falloff solutions thus obtained depends upon the ratio of the sample's pore volume to the volume of the upstream gas reservoir. When this ratio is small, the steady-state solution is nearly exact. As the ratio increases (larger PV or smaller reservoir volume), the correction that must be applied to the steady-state solution progressively increases.

6.4.1.1 Pressure-Falloff, Axial Gas Flow

6.4.1.1.1 Apparatus and Procedure

The pressure falloff apparatus (see Figure 6-22) employs an upstream gas manifold that is attached to a sample holder capable of applying hydrostatic stresses to a cylindrical sample of diameter D and length L. An upstream gas reservoir of calibrated volume can be connected to, or isolated from, the calibrated manifold volume by means of a valve. (Multiple reservoir volumes are used to accommodate a wide range of permeabilities.) The outlet port from the sample holder is vented to the atmosphere. This port is provided with a valve, which can be closed for start-up purposes. An accurate pressure transducer that measures gauge pressure is connected to the manifold immediately upstream of the sample holder.

The reservoir, manifold, and sample—to the outlet valve—are filled with gas. After a few seconds for thermal equilibrium, the outlet valve is opened to initiate the pressure transient. When the upstream pressure has decayed to about 85 percent of the fill pressure, during which time a smooth pressure profile is established throughout the length of the sample, data collection is started. Pressures at selected intervals and corresponding elapsed times are read and recorded.

6.4.1.1.2 Calculations

Procedures for calculating k_∞, b, and β from axial pressure falloff data are given in B.6.8.2.

6.4.1.1.3 Advantages

Advantages of this method include:

a. Slip-corrected permeability, k_∞, Klinkenberg gas slippage factor, b (up to about 1,000 md), and Forchheimer coefficient of inertial resistivity, β, can all be determined from a single pressure falloff test.

b. Non-slip-corrected permeability, k_g, can be calculated (see 6.4.1.1.5) from these results for any desired gas at any mean pore pressure for purposes of comparison with conventional measurements.

c. No flow meters are required for the method. Flow rates are calculated from pressure-time measurements.

d. For a core plug of a given permeability, the time required for pressure to fall from one pressure to another pressure is directly proportional to the volume of the upstream gas reservoir. Therefore, measurement times can be controlled for a wide range of permeabilities by providing several reservoir volumes—small for low permeabilities and larger for higher permeabilities.

e. The practical permeability range is from 0.001 to 30,000 millidarcys if multiple reservoir volumes and transducer ranges are used.

f. Departure from Stokes flow is not a concern. This is accounted for in the data analysis scheme.

g. Slip-corrected permeability measurements at high confining stresses more accurately reflect reservoir conditions, especially for low permeability samples.

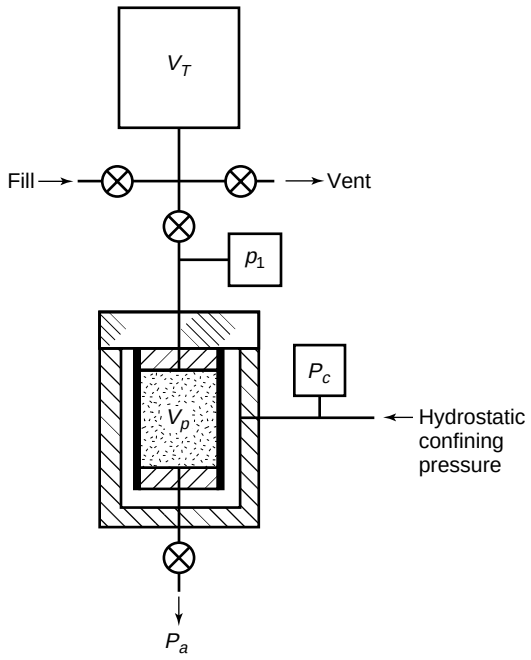


Figure 6-22—Schematic of Pressure-Falloff Gas Permeameter

6.4.1.1.4 Precautions and Limitations

Precautions and limitations of this method include:

a. To minimize thermal effects with high permeability plugs, small fill pressures (<25 psig), small diameter (1 inch), and long lengths (≥2 inches) should be used.

b. To minimize measurement time with low permeability samples, plug diameter should be large (1.5 inches) and its length short (≤1.5 inches).

c. The technique requires high quality pressure transducers, fast data acquisition equipment, and a high computational demand. As a practical matter, therefore, it requires a digital computer.

d. Gas reservoirs must be provided with considerable thermal mass and have high heat transfer capability to avoid work-related temperature changes during gas expansion, especially with high permeability samples. Temperature changes are further minimized by using helium, and by reducing the starting pressure for these falloff tests. High permeability samples also demand large diameter ports in the gas delivery system to minimize internal pressure differentials.

e. Low permeability samples require a leak-tight system (for this or any other technique), and no bypass in the sample holder (past the rubber sleeve). Adequate thermal insulation must be provided to minimize temperature changes induced from ambient changes.

f. Low permeability samples often exhibit extreme stress sensitivity. As mean pore pressure decreases, net stress increases (with a constant confining stress), and permeability decreases. In calculations, permeability is assumed to remain constant during the entire pressure falloff. This assumption can result in the calculation of low or negative β for very stress-sensitive samples. Because the largest changes in permeability occur at low net stresses, stress sensitivity is minimized by making measurements at higher net stresses (>2000 psi), and by reducing the variation in pore pressure during a test.

6.4.1.1.5 Calculation of Gas Permeability From Klinkenberg Permeability

Due to the historical prevalence of steady-state axial gas permeability measurements made without the Klinkenberg correction, huge data bases have been developed from these measurements. It is often desired to compare new data with previous measurements on the same reservoir. To do this, k_∞ must be "uncorrected" for gas slippage. The non-slip-corrected permeability, k_g, for any gas at any desired temperature and mean pore pressure can be calculated from a measured k_∞ and b as follows:

a. Calculate the sample's "gas slippage radius," r_b, (from Equation 13 in 6.2.1.1) using appropriate values of molecular

weight, viscosity, and the b obtained for the gas used in the measurement at the absolute temperature of the measurement.

b. From this r_b, find b from Equation 12 for the new gas, using its molecular weight and viscosity at the desired temperature.

c. From the new b, the measured k_∞, and the desired mean pore pressure, calculate k_g for the new gas from Equation 21.

These steps can be combined into a single equation:

$$k_g = k_\infty \left(1 + \frac{\frac{b_m \mu_c}{\mu_m} \sqrt{\frac{T_c M_m}{T_m M_c}}}{P_{m_c}} \right) \qquad (31)$$

Where the subscript m refers to the measurement gas or condition, and c refers to the desired gas or condition for which k_g is to be calculated. To illustrate the use of Equation 31, suppose that for a particular rock, the measured k_∞ and b are 10.62 millidarcys and 6.57 psi, respectively, using helium at 72°F. For purposes of comparing this measurement with old data taken on the same formation, it is desired to calculate k_{air} at a mean pore pressure of 18.0 psia and temperature of 75°F. The molecular weight of helium is 4.0026, and its viscosity at 72°F is 0.01967 cp. The average molecular weight of air is 29.0, and its viscosity at 75°F is 0.01837 cp. Therefore, from Equation 31, k_{air} at 75°F and 18.0 psia is:

$$k_{air} = 10.62 \left(1 + \frac{\frac{6.57 \times 0.01837}{0.01967} \sqrt{\frac{(75 + 459.7)4.0026}{(72 + 459.7)29.0}}}{18.0} \right) = 11.97 \text{ md}$$

A note of warning: many of the earlier measurements were made at low stress levels, typically at 250 or 400 psi. Even with the above correction, the old k_{air} values on comparable rock may be higher if the new measurements were made at significantly higher stresses. Furthermore, old databases may not include the mean pressure and temperature required to perform these calculations.

6.4.1.2 Pressure Falloff, Probe Permeability Measurements with Gases

The pressure falloff technique is easily adapted for probe-permeameter measurements to provide rapid, non-destructive gas permeabilities that are corrected for both gas slippage and inertial resistance. As with steady-state devices, measurements are typically made at high sampling densities and nearly zero stress on slabbed or unslabbed whole core samples.

6.4.1.2.1 Apparatus and Procedure

Figure 6-23 schematically shows the instrument described by Jones.[22] To operate, the upstream volumes are filled with gas (usually dry nitrogen) to a pressure of about 10 psig. The probe is positioned above a sample, and then lowered and sealed against its surface by a fixed pneumatic pressure on the probe operator piston.

The pressure falloff is initiated by opening the internal valve nearest the probe. The appropriate gas reservoir volume for the particular permeability measurement is selected via internal valves in the manifold. This selection is made on the basis of the rate of pressure decline when all internal volumes are connected to the probe. If the rate is slower than a predetermined value, appropriate valve(s) to the larger tank(s) are closed.

Pressures at preselected intervals and corresponding elapsed times are read and recorded for the calculation of k_∞.

6.4.1.2.2 Calculations

Calculation procedures are discussed in C.6.8.3.

6.4.1.2.3 Advantages

Advantages of this method include:

a. The practical permeability range of this technique is from 0.001 to 30,000 millidarcys.

b. Measurement times in this range vary from 35 to 2 seconds.

c. No flow meters are required. Only time-pressure measurements are made.

d. Both slip-corrected (k_∞) and nonslip-corrected (k_g) permeabilities are obtained from a single pressure falloff test.

e. Inertial flow resistance effects, which can be severe even with small pressure differences and low permeability samples, are eliminated from the calculation of permeability with this technique.

f. The method is nondestructive in the sense that there is no need to cut core plugs. It permits high density sampling in sections that exhibit frequent and severe permeability variation.

g. The volume of rock sample investigated by a single test can be varied somewhat by varying the size of the probe tip seal dimensions. However, in general this volume is quite small (the permeability measured is localized). This statement also applies to steady-state measurements (see 6.3.1.2.3).

6.4.1.2.4 Precautions and Limitations

Except for the problems of inertial resistance and gas slippage, which are handled by the calculation procedures, the precautions and limitations that apply to steady-state probe permeability measurements (see 6.3.1.2.4) also apply to the pressure-falloff measurements.

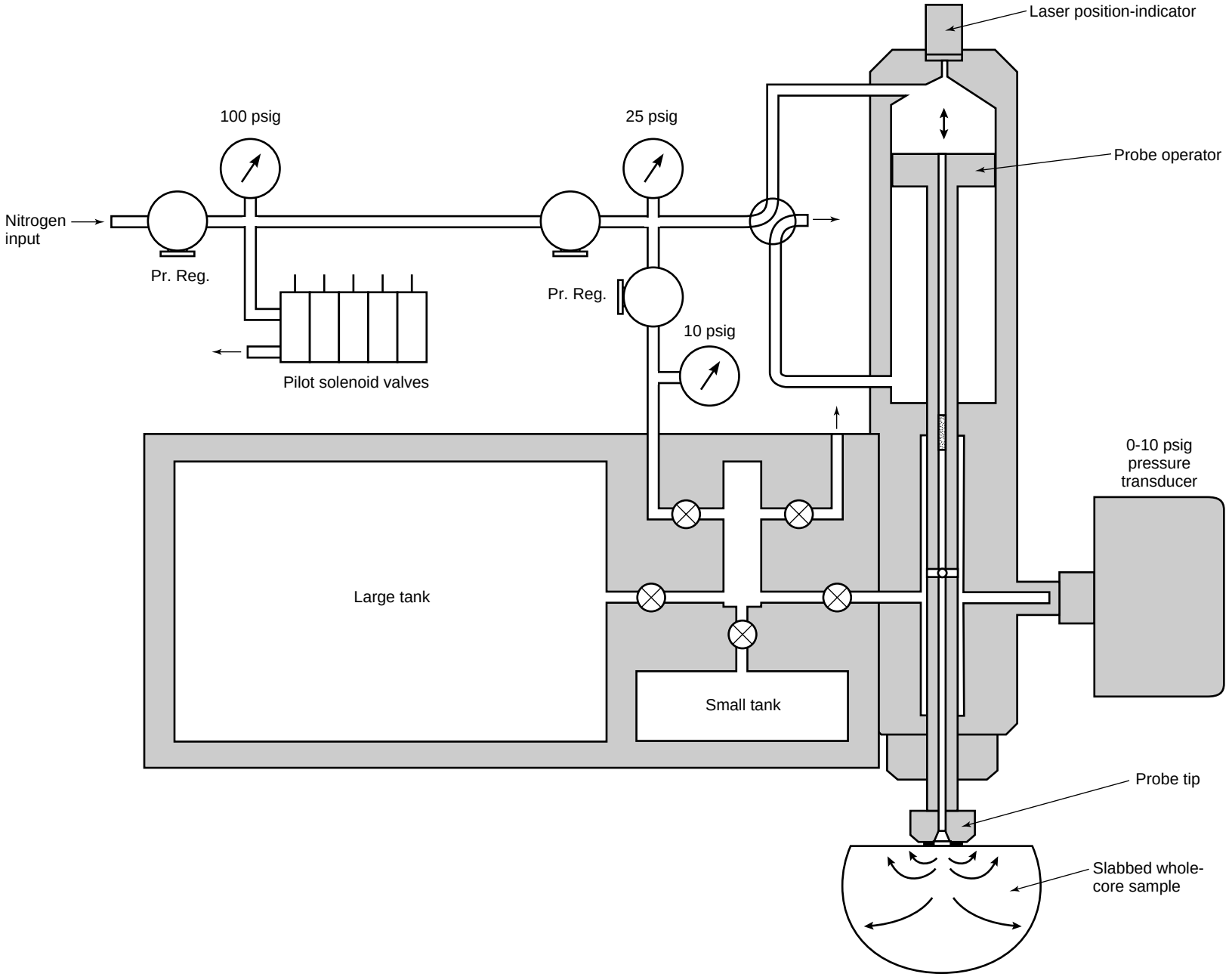


Figure 6-23—Schematic of Pressure-Falloff Probe Permeameter (U.S. Patent 5,237,854)

6.4.1.3 Pulse-Decay, Axial Gas Flow

6.4.1.3.1 Apparatus

The pulse decay apparatus, shown in Figure 6-24, consists of an upstream gas reservoir of volume V_1, a sample holder capable of applying high confining stresses (usually isostatic), which contains the sample of nominal pore volume V_p, and a downstream reservoir of volume V_2. A differential pressure transducer measures the pressure difference between the reservoirs, and a second transducer measures the absolute pressure in the downstream reservoir.

6.4.1.3.2 Procedure

With valves 1 and 2 open, both reservoirs and the sample are filled with gas (usually dry nitrogen) to a pressure typically between 1,000 and 2,000 psig. The fill period must allow adequate time for the gas to diffuse into the (typically) low-permeability sample. After the fill period, the fill valve is closed and the pressure is monitored until no further change is observed, indicating thermal and pressure equilibrium. This stabilized pressure is $P_2[0]$. All valves should be of the type that have no change in internal volume when opened or closed.

After equilibrium, valves 1 and 2 are closed, and the pressure in the upstream reservoir is increased by Δp_1, which is 2 to 3 percent of $P_2[0]$. After the pressure in V_1 becomes stable, i.e., when Δp_1 becomes constant, valve 1 is opened, initiating the pressure-transient portion of the measurement.

6.4.1.3.3 Calculations

See D.6.8.4 for details.

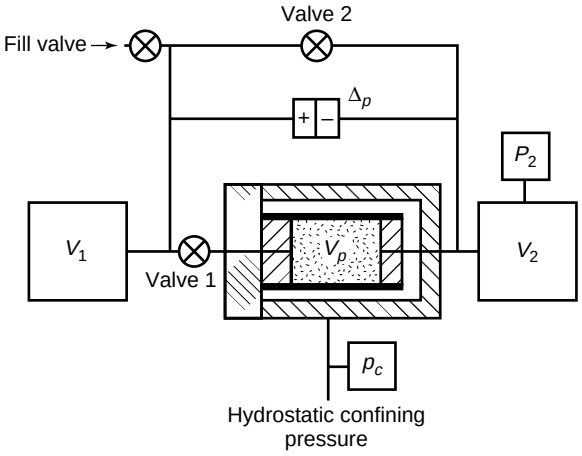


Figure 6-24—Pulse Decay Apparatus for Axial Flow of Gases

6.4.1.3.4 Advantages

Advantages of this method include:

a. The pulse decay technique is applicable to the measurement of very low permeabilities, 0.1 millidarcy to about 0.01 microdarcy, and possibly can be extended in either direction by careful selection of gas reservoir volumes and pressure transducer ranges.
b. No flow meters are required. Only time-pressure measurements are made.
c. These measurements are well adapted for application of reservoir-condition stresses to provide representative permeabilities.
d. Porosity can be measured simultaneously or separately in the same apparatus.

6.4.1.3.5 Precautions and Limitations

Precautions and limitations of this method include:

a. Due to the very low permeabilities that can be measured by this technique, leak-tightness of the apparatus is of ultimate importance. Control of ambient temperature variations is also critical.
b. Even though high back pressures are employed, permeabilities obtained are not corrected for gas slippage and may be somewhat high. For example if b for nitrogen were 100 psi for a particular plug, and the mean pore pressure were 1,000 psia, the gas permeability obtained would be 10 percent higher than k_∞.

6.4.2 Transient Pressure Techniques for Liquids

6.4.2.1 Pulse Decay, Axial Liquid Flow

Unless large pressure gradients are imposed (100 to 1,000 psi/cm or more), liquid permeabilities of tight rocks (k <0.1 millidarcy) are difficult to measure, or are at least time-consuming with steady-state methods. However, permeabilities down to about 0.01 microdarcy can be measured using pulse decay techniques.

Very low flow rates are calculated from rates of pressure change in liquid-filled reservoirs of known compressive storage—as liquid expands from an upstream reservoir or is compressed into one downstream of the sample. Thus, important parameters in these measurements are effective compressive storages of the upstream and downstream reservoirs and of the rock sample. The effective compressive storage of each reservoir is its internal volume multiplied by the sum of the liquid compressibility and the compressibility of the vessel. The sample's compressive storage is the product of its pore volume and the sum of liquid and PV compressibilities. The liquid pulse decay apparatus should have provision for measuring compressive storages of the upstream and downstream volumes (see E.6.8.5.1).

6.4.2.1.1 Procedure

For a pulse decay liquid permeability measurement (see Figure 6-25), a completely liquid-saturated rock sample is loaded into the sample holder, which is then pressurized, preferably to a fairly high (usually hydrostatic) confining stress. The high stress minimizes permeability variation from changes in net stress due to varying pore pressure. The system is filled with liquid, as described in E.6.8.5, and pressurized to the initial pore pressure. The fill valve is left open long enough to allow the high pressure liquid to diffuse into the sample. With valves 1 and 2 still open, the fill valve is closed, and the downstream transducer is monitored until no further change in pressure is observed. Then valves 1 and 2 are closed and the pressure pulse, Δp_1, is generated by screwing in the needle valve. All valves, except for the needle valve, should be of the type that have no change in internal volume when opened or closed.

The pulse decay is initiated by opening valve 1. The initial pressure difference across the sample,$\Delta p[0]$, will be slightly less than Δp_1, which was generated by the needle valve with valve 1 closed, thereby excluding the upstream dead volume, V_d, from the total upstream volume, V_1. Therefore $\Delta p[0]$ must be calculated, as shown in E.6.8.5.

6.4.2.1.2 Indication of Fairly Homogeneous Sample

The upstream pressure decreases while the pressure pulse traverses the length of the sample, but the downstream pressure remains constant until the pulse "emerges." Thereafter, the upstream pressure continues to decrease, and the downstream pressure rises. If the sample is reasonably homogeneous and upstream and downstream compressive storages are equal, then shortly after the pulse-propagation period, the upstream pressure decrease is offset by an equal downstream increase, and the mean pore pressure will remain constant as the $\Delta p[t]$ continues to decrease linearly—in a semi-logarithmic decay.

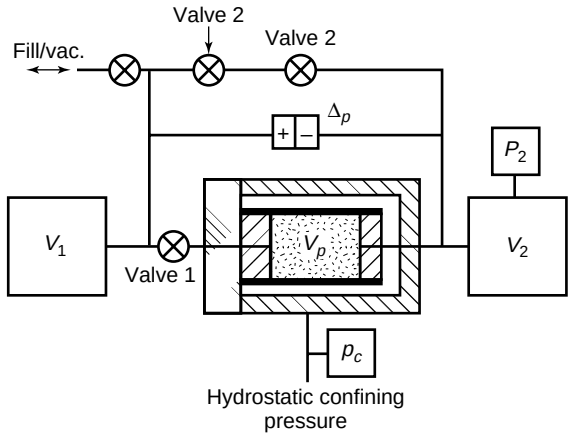


Figure 6-25—Schematic of Liquid Pulse-Decay Permeameter

6.4.2.1.3 Heterogeneous Sample Indication

However, if the sample is not homogeneous, but is characterized by a fracture system of relatively high fluid conductivity and a matrix porosity of low conductivity, and if the compressive storage of the sample is nearly as large, or larger, than those of the upstream and downstream reservoirs (small reservoirs), the mean pore pressure will continue to decrease even after $\Delta p[t]$ has reached zero, and the linearity of the semi-logarithmic pressure decay may have been distorted. In this case $\Delta p[t]$ is fairly quickly dissipated by the fracture system, but liquid continues to diffuse slowly into the tight matrix. If the reservoir storages are large compared to that of the matrix, the mean-pore-pressure decrease, due to liquid movement into the matrix, is barely observable. For a more complete discussion of the measurement of heterogeneity in core samples, see Kamath, et al.[29]

6.4.2.1.4 Calculations

See E.6.8.5.3 for details of calculations for axial liquid pulse decay measurements.

6.4.2.1.5 Advantages

Advantages of this method include:

a. No corrections for slippage are required for liquid measurements.
b. High stress liquid permeability measurements may be more representative of reservoir conditions than gas measurements.
c. This technique is useful for measurement of low permeability rocks (from about 0.01 microdarcy to 0.1 millidarcy).
d. The work of expansion or compression is small in liquid-filled systems. Hence work-related temperature changes are correspondingly small.

6.4.2.1.6 Precautions and Limitations

Precautions and limitations of this method include:

a. Maintenance of isothermal conditions is of utmost importance. Small ambient temperature changes can cause large pressure changes in high-pressure liquid-filled systems.
b. High quality pressure transducers and data acquisition system are required.
c. A leak tight system is essential for high pressure measurements of low permeability plugs.
d. The usual precautions of corrosion resistance, fluid compatibility, careful filtration of liquids, and exclusion of gas from the system must be observed.

6.5 ACCURACY AND PRECISION

6.5.1 Part 1

6.5.1.1 Introduction

Permeability data represent quantitative measurements, and correct values are important. All analytical methods are subject to random scatter and to operator, equipment, and technique-related errors, which, taken together, comprise experimental uncertainty. To distinguish between a real trend in a data set and variation due to experimental uncertainty, it is recommended that core analysis data be reported together with a statement of the uncertainty with which these data were recorded.

6.5.1.2 Definitions

6.5.1.2.1 precision (or reproducibility) of a measurement: An expression of the limits within which the measurement, with a stated probability, can be reproduced provided that the measurement is subject only to stochastic variation. Precision is determined from repeated measurements on a single sample, and is normally given as a standard deviation or relative standard deviation, also known as the coefficient of variation.

6.5.1.2.2 accuracy of a measurement: An expression of the closeness of agreement between the experimentally obtained value and the true value. The difference between the measured and the true value is called the error. Results falling outside the maximum allowable error or tolerance may be caused by operating errors or systematic errors.

6.5.1.2.3 bias (or systematic) error: A deviation that persists during a measurement cycle and affects all measurements in the same way. Bias is normally due to instrumental drift, or faulty calibration, or operating procedures.

6.5.1.2.4 applicability: The concept of applicability, in addition to precision and accuracy, is appropriate here. Permeability measurements may have been performed with high precision and accuracy, producing results close to true values of permeability for the reported conditions under which they were measured. Yet, these precise, accurate values might be quite different from those that would be obtained for conditions that exist in the formation from which the samples were taken. Reasons for differences include different stress conditions, rock-fluid interaction, differences in pore pressure, or gas composition if gas slippage is ignored, etc.

6.5.1.3 Error Statements in Permeability Measurements

The concept of accuracy requires that the true value of a sample be known. This is usually not the case. Instead certified standard reference materials for scientific or industrial use are obtained from recognized standard institutions. These materials are analyzed at regular intervals together with routine samples. Accuracy is then obtained by comparing the mean value, calculated from repeated analyses on the standard material, with the certified value for that standard.

In permeability measurement, suitable standard materials could be natural rocks or permeable synthetic materials. However, synthetic samples cannot yet be produced in a way that allows an accurate pre-determination of permeability. Therefore, each standard must pass an extensive testing program before it is certified, and afterwards there is risk that the standard will change during regular handling in the laboratory. So far, no certified permeability standards have been available from recognized standard institutions. Therefore, a different approach for determining accuracy in permeability measurement is recommended.

6.5.1.4 Internal Standards

The concept of internal permeability standards is introduced for determining accuracy, and as a facility in testing equipment for bias or systematic errors. Internal standards can be synthetic and/or natural samples covering a large range of permeability values, which have been analyzed repeatedly by different operators on different (carefully calibrated) apparatus using the required fluid. When many measurements have been collected, a mean value and standard deviation is calculated for the sample. If the variation about the mean is small, i.e., the standard deviation is low, the sample is accepted as an internal standard, and can be used routinely to check accuracy of equipment and correct for bias in the results.

Synthetic porous and permeable media are available in different materials, e.g., as porous ceramics and glasses, and as sintered plastic, glass, or metal beads. These materials are often supplied from the manufacturer with a specified permeability value, which, however, should only be regarded as approximate.

6.5.1.5 Methodological Errors

All equations for calculating permeability presented herein assume fluid flow through homogeneous, isotropic samples, under isothermal conditions. Thus they define an average or effective permeability in the overall direction of flow dictated by the experiment (i.e., axial, radial, transverse, etc.). Unless the internal, macroscopic structure of each sample is determined by independent means, it is virtually impossible to model the flow otherwise. Because real rocks are almost *never* isotropic or homogeneous, the assumption that they are will invariably distort actual flow patterns, and consequently, the calculated permeability, and also inertial resistivity and gas slippage factor, whenever they apply.

Equations for unsteady-state analyses require correction for nonconstant mass flow (with distance from the injection point) at any instant during the pressure transient. Therefore,

a sample's pore volume must be determined, either independently or in conjunction with the permeability measurement itself. Inaccuracies in the estimation of pore volume will lead to permeability errors, especially when *PV* is nearly as large, or larger than the gas reservoir volume. Small *PV* errors are inconsequential when reservoir volumes are large on a relative basis.

Furthermore, the transient techniques assume constant permeability throughout the duration of a test. Changes in pore pressure change the net stress to which the sample is subjected, thereby slightly changing its permeability during a test. In a pressure falloff measurement, this can distort the calculation of inertial resistivity (β). These errors are minimized by limiting the magnitude of the pressure falloff, and by increasing the confining stress (where changes in net stress have less effect on permeability—per Figure 6-9).

Serious errors can result when operating conditions are such that Darcy's law does not apply because of significant inertial resistance. These are most prevalent with steady-state, axial flow gas measurements on high permeability plugs (several darcys), or steady-state probe permeability measurements on samples of about 10 millidarcys or more. These errors are minimized by using very small values.

Other errors can be introduced by improper sample preparation techniques, such as inadequate cleaning, or nonremoval of salt crystals from extracted samples that previously contained nearly saturated brine. Improper drying of samples with high clay content can either enhance or reduce permeability. Embedded fines from cutting or grinding the ends of samples can reduce permeability. High points on nonsquare ends of a core plug can be crushed at high axial stresses, altering permeability. Incompatible or dirty liquids cause plugging. High velocity fluids (especially liquids) can cause fines migration and plugging.

Gas permeabilities (uncorrected for gas slippage), measured at low confining stress, if properly reported (including the gas used, mean pore pressure, and net stress), are properly specified and do not per se constitute an error. However, if they are expected to predict reservoir performance accurately without correction, they may not be particularly applicable, especially for lower permeability samples, where the corrections are substantial.

6.5.1.6 Equipment Component Errors

Main error sources associated with test equipment are leaks, sample bypass, pressure transducer error, flow meter error, and ambient temperature variation. Additionally, nonisothermal fluid expansion, inaccurate calibration of reservoir volumes, and inadequate data acquisition resolution and speed can cause errors in unsteady-state measurements. With probe permeameters, probe positioning accuracy and sealing force repeatability are also important.

6.5.1.6.1 Leaks and Sample Bypass

All types of permeameters are subject to leaks in tubing, fittings, valves, transducers, flow meters, and core holder. Bypass of fluid between a core plug and rubber sleeve of the sample holder, or between a sample and the probe seal of a probe permeameter, also constitutes a leak. The magnitude of error caused by a leak generally increases as permeability decreases. For example, a small leak in the measurement of a 3 darcy plug would barely affect its calculated permeability, whereas the same leak rate with a 0.001 millidarcy plug would be disastrous.

Large leaks can be located in gas permeameters with the aid of a soap solution that forms bubbles when applied to a leak. Small leaks are most easily located by means of pressure measurements. Various portions of the flow system are pressurized with gas, then monitored by the system's pressure transducer. The leak is located through a process of elimination. It may be necessary to load an impermeable steel plug in the sample holder to isolate the upstream and downstream portions of the apparatus. Some systems incorporate automated leak-detection routines.

Sample bypass usually can be eliminated by increasing confining stress and/or reducing the hardness (durometer) of the rubber.

6.5.1.6.2 Pressure Transducer Error

Accuracy of a pressure transducer is usually specified as the root sum of squares (RSS) of deviations of its output from actual pressures throughout its range as a percent of its full scale (FS) reading. RSS accuracies of 0.5 percent FS or better are generally available. Most transducers are provided with at least two adjustments—zero and span. The zero adjustment is an offset to permit zero electrical output at zero applied pressure. The span adjustment changes the transducer's gain or multiplier that converts its electrical output to a pressure. Some transducers also have one or more internal linearity adjustments. Pressure transducers are also affected by temperature changes, and are provided with temperature compensation. Price is often nearly proportional to the quality of the temperature compensation.

The user should keep in mind consequences of the "full scale accuracy" specification. For example, a 0.5 percent transducer, used at 10 percent of full scale, can have a 5 percent error in the value of its pressure reading, and be within specification. In view of the RSS definition, the particular error at a given point could be greater than the given RSS percent of the full scale reading.

With proper calibration techniques that allow for frequent mathematical rezeroing capability and nonlinear curve fitting, as outlined in 6.61, a transducer's repeatability is among its most important characteristics. High quality transducers with 0.01 or 0.02 percent FS repeatability are available. To minimize hysteresis effects, a transducer should always be cali-

brated in the direction of its anticipated use. For example, for pressure falloff measurements, a transducer should be calibrated to its full scale pressure first, followed by a sequence of descending pressures.

Unsteady-state techniques place extra demands on pressure measurements. Pressure-time derivatives are calculated. Therefore noise, or small voltage fluctuations must be minimized, and resolution of the reading must not be truncated by an analog-to-digital voltage converter (ADC) with too few bits. Noise is minimized by starting with a good quality transducer with low noise output and shielded cables. An analog, low-pass filter can be used in conjunction with digital filtering. With a reasonably fast ADC (1,000 conversions per second) multiple readings can be taken, then averaged. A slow, integrating ADC (60 conversions per second) performs averaging automatically. A 15 bit ADC (0.0031 percent of full scale resolution) minimum is recommended for unsteady-state measurements to minimize truncation error.

High quality timer cards are available, with accuracies in the part per million range. It is imperative to query the timer card immediately either before or after reading the ADC, with no intervening commands in the operating program, so that pressure intervals correspond exactly to time intervals for the calculation of pressure-time derivatives. Furthermore, pressure intervals must be sufficiently large that noise in the measurements does not destroy accuracy of the derivative calculation. Optimum intervals may have to be determined by trial and error for a particular system.

A non-smooth plot of pressure-time derivatives vs. geometric mean pressures from pressure falloff data indicates either excessive noise or truncation of time or pressure readings. The problem is usually eliminated by increasing the pressure and/or time intervals, increasing the resolution (number of bits) of the ADC, or by improving the quality of the transducer. The sampling routine should be written in assembly language for maximum speed.

6.5.1.6.3 Flow Meter Error

Steady-state techniques depend on accurate flow rate determination. Direct reading volumetric devices in combination with time readings can yield accuracies of better than 1 percent. Indirect devices (calibrated orifices, mass flow meters and controllers) can normally be adjusted to yield a precision better than 1 percent, but accuracy is totally dependent upon calibration. Several flow meters are required to cover the entire permeability range. Each one should be used only within its linear working range. See 6.6 for mass flow meter calibration.

6.5.1.6.4 Ambient Temperature Variation

Changes in temperature affect both volumetrics and fluid viscosity. Temperature should be kept constant during a permeability measurement. Never place the apparatus near a heating or cooling vent or near a window where the sun's radiation can cause large temperature changes. In unsteady-state equipment, the rate of temperature change within fluid reservoirs must be such that temperature-induced pressure changes are small relative to pressure decreases that are caused by flow of fluid from a reservoir.

In field applications of probe permeameter measurements, temperature changes affect fluid viscosity, flow rate and pressure measurements.

6.5.2 Part 2

6.5.2.1 Error Statements in Reporting

Presentation of measured data with associated error limits in a core analysis report can be based on two different approaches: (a) measured values are used to calculate and report a mean value with associated error limits as calculated below, and (b) measured values are given together with a statement of accuracy, which the laboratory can base on repeated measurements of internal standards.

6.5.2.1.1 Calculated Error Limits

Suppose that a large number of repeated measurements (normally more than 100) are performed on a single sample. If the measurements are only subject to stochastic variation, it is generally believed that data have a normal or gaussian distribution with a mean value, $\bar{X}$:

$$\bar{X} = \frac{1}{n}\sum_{i=1}^{n} X_1$$

and standard deviation, *s*:

$$s = \sqrt{\frac{\sum_{i=1}^{n}(X_i - \bar{X})^2}{n-1}}$$

Where $(X_i - \bar{X})$ is the deviation of a single measurement from the mean and n is the total number of measurements. The result is reported as $\bar{X} \pm s$. The standard deviation is a measure of the spread or variation of data about the mean. A normal distribution has a probability that 68.3 percent of the observations will fall within the limits of plus or minus one standard deviation from the mean. The coefficient of variation, *CV,* is the relative standard deviation expressed as a percentage:

$$CV = 100(s/\bar{X})$$

Often, only a limited number of measurements are recorded. This means that we are dealing with small sample statistics and the *t*-distribution should be applied. Often it is more relevant to define confidence intervals about the mean than to quote the deviation. The confidence interval is an esti-

mate of the limits within which the true value lies with a stated probability. When reporting core analysis results it is therefore recommended to quote results as:

$$\bar{X} \pm t(s/\sqrt{n-1})$$

which is the confidence interval. Values of t are found in most statistical textbooks or in statistical tables, e.g., Fisher and Yates[31]. These values are given for various "degrees of freedom," n–1, and for different "confidence levels" or probabilities. A 95 percent confidence level is often used (corresponding to standard deviations, adjusted for the small number of repeat measurements).

The following example illustrates these concepts. Three repeat measurements were made on a sample. Calculated permeabilities are 3.35, 3.18, and 3.42 millidarcys, from which the mean and standard deviation are calculated:

$$\bar{X} = 3.32 \text{ md}$$

$$s = 0.12 \text{ md}$$

The value of t for 2 degrees of freedom (= n–1, where n = 3 measurements) and a 95 percent confidence level is 4.303. Therefore the confidence interval is:

$$3.32 \pm 4.303(0.12/\sqrt{3-1}) = 3.32 \pm 0.37 \text{ md}$$

In other words, these calculations imply that with a probability of 95 percent, the true permeability lies within the interval from 2.95 to 3.69 millidarcys, provided that the measurements were not biased.

Klinkenberg permeability is calculated from gas permeability measurements by performing linear regression on permeability vs. reciprocal mean pore pressure values. The goodness of fit of the regression line can be assessed from the coefficient of correlation, r, which should be reported with the permeability value. If we still want to have a confidence level of 95 percent, r must be greater than or equal to 0.95 for a 4 point regression line, and at least 0.997 for a 3 point line. Samples giving values of r lower than these should be inspected for fractures, vugs, or other irregularities, and if present this should be reported in the data listing. Also the measurements should be checked for the possibility of non-negligible inertial resistance at the higher mean pore pressures.

6.5.2.1.2 Laboratory Reported Error Limits

In routine core analysis, a large number of determinations on internal standard plugs will be accumulated over a period of time. It is valid that a laboratory prepare a table showing the accuracy and precision obtained for different permeability ranges based on standard measurements.

An impression of the accuracy that can be obtained in permeability measurements was reported by Thomas and Pugh[32]. Using conventional steady-state measurements performed by many laboratories all over the world on sets of standard plugs, they quoted the statistically derived confidence intervals with a probability (confidence level) of 99 percent from which the other confidence intervals were calculated in the table below.

	Confidence interval, %, with probability of:		
k_g range, millidarcys	68.3% (= mean CV)	95%	99%
0.01-0.1	±8%	±16%	±21%
0.1-1.0	8	16	21
1-50	5	10	13
50-1,000	3	6	8

The numbers shown in this table can be interpreted as follows. Suppose a certain core plug has a true permeability of 10.0 millidarcys, which lies in the 1-50 range above. Thus, if a total of one thousand measurements were made on this plug by laboratories throughout the world, approximately 683 of the reported permeabilities (68.3 percent of the total) would be expected to lie within the range from 9.5 to 10.5 millidarcys (±5 percent of the true value). Similarly 950 of them would be expected to lie within 9.0 to 11.0 millidarcys, and 990 within the range from 8.7 to 11.3 millidarcys (with 10 measurements lying outside this range).

6.5.2.1.3 Number of Significant Figures

The number of figures given in a report should respect the error associated with the measured value. For example, 4.2357, ±0.0327 md should be reported as 4.24, ±0.03 md; i.e., the error is understood to occur on the last decimal in the rounded number. In view of the table above, probably only two significant figures are justified (i.e., 4.2 md), unless a particular laboratory produces significantly better results than those upon which the table is based.

6.6 CALIBRATION OF INSTRUMENTS

6.6.1 Pressure Transducer Calibration

This discussion applies to a gauge pressure transducer, which should read zero when it is exposed to ambient atmospheric pressure. It also applies to an absolute transducer, provided that a high vacuum is applied for "zero (absolute) pressure" readings. Any pressure transducer must be calibrated periodically. Even though a transducer is normally calibrated at the factory within stated tolerances, it should be recalibrated in its flow system configuration, with the system's analog-to-digital converter (ADC). This is accomplished with a high quality primary standard, such as a dead weight tester or precision manometer and cathetometer, or with a secondary standard pressure transducer (preferably

accurate to 0.01 to 0.02 percent full scale), traceable to a primary standard. A known pressure is applied, and the resulting voltage or ADC output is read. This is repeated at several, nearly equally spaced intervals throughout the range to be calibrated, including zero pressure. A 21 point calibration (intervals of 5 percent of full scale), minimum, is desirable.

6.6.1.1 Calculations

These data (if reasonably linear—to within 0.1 to 0.5 percent of full scale) are fit with a polynomial, using least squares techniques. A good rule of thumb for choosing the polynomial order is: the number of data points taken in the calibration must be at least double the number of coefficients calculated from the polynomial fit. For 21 points or more, a fourth-order polynomial is a good choice.

In general, it is nearly impossible to set the transducer's zero-adjust potentiometer so that the transducer outputs exactly zero voltage at zero pressure at the time of calibration. Furthermore, some ADCs read only positive voltages (negative voltages are output as zero). For these ADCs, the transducer's zero offset must be sufficiently positive that zero drift will never cause a negative zero-pressure voltage. A fourth-order polynomial, with which calibration data are fit, has the form:

$$p = a_0 + a_1 v + a_2 v^2 + a_3 v^3 + a_4 v^4 \quad (37)$$

Where p is the actual pressure (determined from the standard), and v is the voltage read from the transducer for each data point. If the zero-pressure voltage is positive, then a_0, from the least-squares fit, will be negative.

It is critical to have calibrations that pass through zero at zero pressure. To illustrate, suppose that a zero-pressure voltage shift of 0.5 percent of the full scale voltage occurred—with time and temperature changes—from the zero-pressure voltage at the time of calibration. This magnitude of zero shift is not uncommon, and does not appear to be too bad. However, even with a perfect span adjustment, this represents a 5 percent error for a pressure reading at 10 percent of full scale, or a 25 percent error for a pressure reading at 2 percent of full scale.

To avoid this problem, operating software should have provision to "mathematically zero" all transducers. Unless a_0, from the calibration, is identically equal to zero in Equation 37, zero drift cannot be corrected by simply subtracting the current zero-pressure voltage from all subsequent voltage readings, then using these voltage differences in Equation 37. Instead, Equation 37 must first be mathematically transformed to:

$$p = A_0 + A_1(v - v_0) + A_2(v - v_0)^2 + A_3(v - v_0)^3 + A_4(v - v_0)^4 \quad (38)$$

To accomplish this transformation, the "best fit" zero-pressure voltage, v_0, at the time of calibration, must be found. This voltage is calculated from all points in the calibration, not just the value read when zero pressure was applied. A first estimate is calculated from:

$$v_0 \approx -a_0 / a_1 \quad (39)$$

This value of v is inserted into Equation 37 to calculate the "best fit" pressure (p_0) that corresponds to this voltage. If the value of v_0 were correct, then p_0 would be exactly equal to zero. The estimate is refined using:

$$v_0[new] = v_0[old] - p_0 / a_1 \quad (40)$$

This new value of the zero-pressure voltage is inserted into Equation 37 to calculate a refined value of p_0. Then these values of v_0 and p_0 are used in Equation 40 to produce a further refinement of v_0. This process is continued until $p_0 \leq 1 \times 10^{-10}$ psig.

Next, the coefficients $A_0 \ldots A_4$ are determined by a least-squares fit of the original calibration data using Equation 38 with the final value of v_0 as determined above. Equations 37 and 38 yield exactly the same calibration, except Equation 38 (after its coefficients have been determined) permits elimination of zero drift by inserting the current value of v_0.

The coefficients $A_0 \ldots A_4$, for a particular transducer, must be installed in an appropriate file in that transducer's operating program. The current value of v_0 must be periodically determined by the operating program of a particular instrument. It is found by pressurizing the transducer to its maximum pressure normally used in the instrument. It is then vented to atmospheric pressure and allowed to stabilize (usually for 30 seconds), after which v_0 is read and stored for subsequent computations with Equation 38.

The fit by a fourth-order polynomial reduces the average absolute deviation by a factor of 4 to 20, compared to that of a *linear* least-squares fit. Equation 38 also eliminates zero drift. The overall improvement (especially at low pressures) is significant compared to a calibration that has not been corrected for zero drift. The coefficient A_0 in Equation 38 should theoretically be equal to zero. It is usually less than 1×10^{-8}, and is calculated and reported only to assure that the transformation of Equation 37 to Equation 38 was successful. For purposes of calculating pressure from an ADC-output, A_0 should be deleted from Equation 38.

6.6.2 Calibration of Mass Flow Meters

Gas mass flow meters respond to mass flow rate, not volumetric flow rate. The volumetric flowrate at some reference condition of temperature and pressure (usually the current ambient temperature and atmospheric pressure), of gas flowing through the mass flow meter, is measured by some independent device (such as a soap bubble meter for low flow rates, which is connected in series with the mass flow meter).

Alternatively, a floating piston with mercury seals in a precision bore volumetric displacement device may be used. Voltage (or some scaled reading) from the mass flow meter is often calibrated against this volumetric flow rate, thereby providing an equation from which volumetric flow rate can be calculated from a voltage acquired. This is not the correct procedure, and unnecessarily limits the calibration! This volumetric flow rate calibration is valid only if the mass flow meter is used at exactly the calibration temperature and pressure. Changes in either pressure or temperature cause errors in the calibration, which could be avoided by simple procedural changes.

Because the mass flow meter's output is proportional to the mass flow rate of a gas, the volumetric flow rates through the bubble meter during a calibration must be multiplied by the absolute pressure in the standard meter (not in the mass flow meter). This is normally the current atmospheric pressure, which can be read from a good quality mercury barometer, with appropriate corrections. This pressure must be converted to the same units that are used in the calculation of permeability (e.g., psia, Pa, or atm). In addition, the volumetric flow rates must be divided by the absolute temperature, (459.67+°F), or (273.15+°C), in the standard meter, and by z, the gas deviation factor, calculated at the temperature and pressure within the standard meter. We will define the result of this manipulation as the "mass flow factor," y_{mf}:

$$y_{mf} = \frac{qP_{cal.}}{z_{cal.}T_{cal.}} \tag{41}$$

The subscripts, *cal.*, above refer to the absolute pressure and temperature that correspond to the volumetric flow rates measured during the calibration.

The advantage of this procedure is that the calibration is not restricted to exactly the same run-time pressure and temperature. Except for small instrumental shifts, the mass flow meter could be operated at any pressure and temperature within its specifications. This often, for example, permits the flow meter to be used at upstream conditions. Upstream operation has advantages: (a) no sand or grit from the core plug enters the flow meter, degrading its accuracy, (b) there is virtually no flow resistance between the mass flow meter and the core plug, allowing faster attainment of steady-state conditions.

Because of instrumental shifts, the mass flow meter should be calibrated at several different pressures and temperatures for most accurate results. Remember that, even though calibrations are reported for conditions in the mass flow meter, the pressure and temperature in the bubble meter (or other volumetric device) are used to calculate the mass flow factors. Normally, effects of temperature and pressure changes are small, and can be interpolated easily.

6.6.2.1 Calculations

For purposes of fitting calibration data, the same procedure should be used as described for pressure transducers, to produce a calibration equation that can be corrected for zero drift. In Equations 37, 38, and 39, the p and p_0 are replaced by y_{mf} and y_{mfo}, respectively. If only 6 to 8 flow rates are calibrated at each pressure, then the fourth-order polynomial should be replaced by a second order polynomial. A minimum of 11 equally spaced points should be obtained for the fourth-order polynomial. Whatever polynomial is used, it should be carefully tested by calculating 50 to 100 points over the entire range to make sure that the final equation does not produce unexpected results between calibration points.

Voltage (or the instrument's reading) at zero mass flow rate is determined at run time by closing valves on both the upstream and downstream ends of the mass flow meter (at its current operating pressure) and ensuring that there is absolutely no flow through it. After 30 seconds for stabilization, output from the ADC, v_0, should be recorded and subtracted from the flowing voltage output, in the flow-equivalent of Equation 38.

Mass flow factors are used in the calculation of gas permeability as follows:

$$k_g = \frac{29392\mu z_m y_{mf} T}{G_f(P_1 - P_2)(P_1 + P_2)} \tag{42}$$

Where:

k_g = non-slip-corrected gas permeability, millidarcys.
μ = gas viscosity, cp.
z_m = gas law deviation factor, calculated at the mean pore pressure and current temperature.
y_{mf} = mass flow factor, (cm^3/s)(psia/°R), or (cm^3/s)(psia/°K).
T = absolute temperature at run time; same units as $T_{cal.}$, °R, or °K.
G_f = dimensional geometric factor for a particular flow configuration, cm.
P_1 = absolute upstream pressure, psia.
P_2 = absolute downstream pressure, psia.

In other words, $y_{mf}T$ is used in place of $q_r P_r / z_r$ in the calculation of permeability. If other units than the above are used in Equation 42, the 29392 is replaced by $2C_2 / C_1$ (see Table 6-1).

6.6.3 Reference Volume Calibration

Unsteady-state gas permeameters contain gas reservoir volumes and dead volumes that must be accurately calibrated. One convenient method is to use gas expansion in conjunction with Boyle's Law, using seven or eight cylindrical steel plugs, each of which contains an accurately-measured axial hole that extends through its entire length. The length and diameter of each hole should be measured to the nearest 0.0001 inch. These plugs must fit inside the sample holder, and must be tightly sealed by the holder's rubber sleeve. The

holes should range in volume from nearly zero to about the volume of the reservoir to be calibrated. The hole volume changes, from one plug to the next, should all be approximately equal. The pressure transducer must be carefully calibrated prior to the volumetric calibration, and it must read zero at zero psig. Ambient temperature changes during the volumetric calibration should be minimized.

6.6.3.1 Calculations

Because the configuration of each system may vary slightly, only the principles of a Boyle's Law calibration will be illustrated. Boyle's Law states that the total of the masses of gas in each part of the system before an expansion is equal to the total mass after the expansion; i.e., no gas enters or leaves the system. Under isothermal conditions, the group (VP/z) is proportional to the mass of gas in volume V, where P is the absolute pressure, and z is the gas deviation factor at the pressure and temperature of the gas contained in volume V.

Referring to Figure 6-26, suppose that one of the several steel plugs has been loaded into the sample holder and is sealed by its rubber sleeve. Then the gas reservoir of volume V_0, that includes the bore of the isolation ball valve; the upper dead volume V_1, which includes the volumes inside the transducer and upper end plug; the known hole volume in the steel plug, V_H; and the lower dead volume, V_2, are filled with helium or nitrogen to pressure p_1 (a gauge pressure). For the most accurate work, this gas should be vented from the system to remove air. The fill-vent cycle should be repeated, and the system filled a third time. Then the isolation valve is closed, and V_0 is vented to atmospheric pressure, after which the vent valve is closed. After pressure equilibration, the final pressure, p_1, is read and recorded.

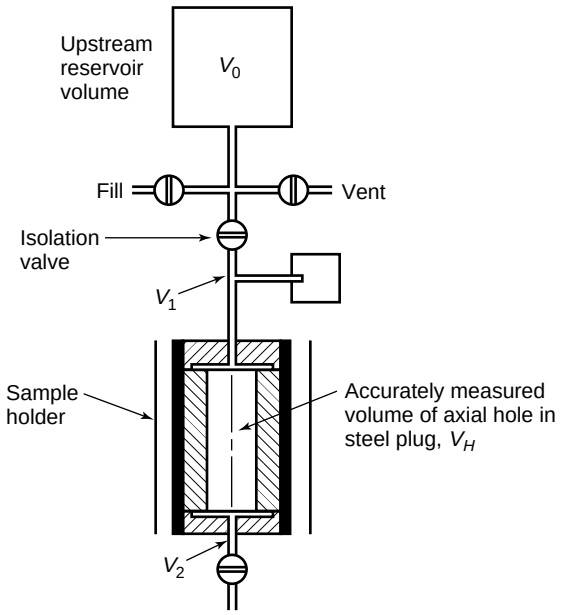


Figure 6-26—Schematic for Calibration of Volumes in Unsteady-State Permeameters

The isolation valve is now opened, expanding gas from the lower portions of the system into V_0. After pressure equilibrium is attained, the final pressure in all parts of the system, p_2, is read and recorded. Each of the remaining steel plugs is loaded into the sample holder and the procedure repeated. Atmospheric pressure, p_a, must be read from an accurate mercury barometer with appropriate corrections.

Now the mass of gas in each part of the system before an expansion is entered on the left hand side of the equation below, and the masses after the expansion are on the right hand side:

$$(V_1 + V_2 + V_{H_i})\left(\frac{p_1 + P_a}{z_1}\right) + (V_0)\left(\frac{P_a}{z_a}\right) = (V_1 + V_2 + V_{H_i} + V_0)\left(\frac{p_2 + P_a}{z_2}\right) \quad (43)$$

A similar equation is obtained for each of the steel plugs. These equations are rearranged to:

$$V_{H_i} = V_0\left[\frac{\frac{p_2 + P_a}{z_2} - \frac{P_a}{z_a}}{\frac{p_1 + P_a}{z_1} - \frac{p_2 + P_a}{z_2}}\right]_i - (V_1 + V_2) \quad (44)$$

The volumes V_0 and $-(V_1 + V_2)$ are now found as the slope and intercept, respectively, from linear regression, where V_H is the y-variable, and the quantity inside the brackets is the x-variable for each plug.

The upper dead volume, V_1, is found by inserting a solid steel plug into the core holder. Gas is expanded from this volume into the now known volume, V_0. An equation similar to Equation 43 is written for the new configuration, and solved for V_1. The reservoir volume, V_T, for a transient permeability measurement is equal to V_1 when the isolation valve is closed, or to $(V_0 + V_1)$ when it is open.

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6.8 APPENDICES

A.6.8.1 Derivation of Equations to Estimate the Maximum Δp for Darcy's Law to Apply

The "condition of Stokes flow," which is a requirement for the legitimate use of Darcy's equation, is met when inertial flow resistance is negligible relative to viscous flow resistance. The purpose of this appendix is to present equations for estimating the maximum allowable Δp to be used in axial flow permeability measurements. If this maximum is not exceeded, Darcy's equation can be safely used in most instances. Equations will first be derived for gases, followed by an equation for the flow of liquids.

A.6.8.1.1 Gases

When Equation 17 (see 6.2.1.2), the differential Forchheimer equation for steady-state flow of gases, corrected for gas slippage, is integrated with respect to length it becomes:

$$\frac{C_1 \Delta p}{L} = \frac{C_2 \mu P_r q_r z_m}{A k_\infty z_r (P_m + f_{Fo} b)} + \frac{C_3 \beta M P_r^{\,2} q_r^{\,2} z_m (P_m + b)}{A^2 R T z_r^{\,2} P_m (P_m + f_{Fo} b)} \quad \text{(A-1)}$$

When the second term on the right hand side is small relative to the first term, f_{Fo} is virtually equal to 1.0. If we assume this value, set all gas deviation factors to 1.0 (which is a good assumption for low pressures), and choose the arithmetic mean pressure, P_m, to be the reference pressure, P_r, for which q_r is calculated, then Equation A-1 simplifies to:

$$\frac{\Delta p}{L} = \frac{C_2 \mu \, v_m}{C_1 k_\infty \left(1 + \frac{b}{P_m}\right)} + \frac{C_3 \beta M P_m v_m^{\,2}}{C_1 R T} \quad \text{(A-2)}$$

where v_m, the Darcy velocity, is equal to q_m / A. This can alternatively be written:

$$\frac{\Delta p}{L} = \frac{C_2 \mu v_m}{C_1 k_\infty \left(1 + \frac{b}{P_m}\right)} \left[1 + \frac{C_3 k_g \beta M P_m v_m}{C_2 \mu R T}\right] \quad \text{(A-3)}$$

In the numerator of the second term inside the brackets, k_g replaces the group $k_\infty(1 + b / P_m)$, per Equation 21. This entire second term is dimensionless, and can be thought of as a "Reynolds number for flow through porous media." Ruth and Ma[33] suggest that this number be called the "Forchheimer number:"

$$N^*_{Fo} = \left(\frac{C_3}{C_2}\right)(k_g \beta \phi)\left(\frac{M P_m}{R T}\right)\left(\frac{v_m}{\phi \mu}\right) \quad \text{(A-4)}$$

The factors on the right hand side of Equation A-4 are grouped into the various components of the Forchheimer number, which is the ratio of inertial to viscous resistance. The first factor contains constants (see Table 6-1) to make the dimensions consistent. The next factor is the characteristic length, analogous to pipe diameter in a Reynolds number for fluid flow through pipes. The next factor can be recognized as gas density at the mean pore pressure. The final factor contains fluid velocity divided by its viscosity. The velocity is obtained by dividing Darcy velocity by rock porosity. Thus it is based upon the average actual area available for flow, not the superficial area. The product of this velocity and gas density is the mass flux of gas through the plug. The porosity that was included in the characteristic length cancels porosity in the final factor.

Unlike pipe flow, where there is a sudden transition from laminar to turbulent flow over a narrow range in the Reynolds number, inertial resistance associated with flow through porous media increases gradually and smoothly as the Forchheimer number increases. This difference can be explained

easily. In laminar pipe flow, fluids are in parallel, streamline flow with virtually no accelerations. Suddenly, at some critical velocity the flow becomes unstable and suffers numerous changes in direction, thus undergoes many accelerations. By contrast, flow through a porous medium is tortuous at any velocity, and involves many accelerations, which increase in magnitude smoothly as mass flux increases. Also, there is a whole spectrum of equivalent pipe sizes in a porous medium. Therefore, sudden transitions from laminar to turbulent flow, with a large increase in the pressure gradient, are not observed.

When the Forchheimer number is zero, all flow resistance is due to viscous shear, and Darcy's law is completely applicable. When it is equal to one, half of the total flow resistance is due to viscous shear. If the Forchheimer number is nine, only 10 percent of the total flow resistance is due to viscous resistance, and the other 90 percent to inertial resistance.

An asterisk is used in Equation A-4 to differentiate the Forchheimer number from the one defined by Equation B-5, which contains k_∞ instead of k_g. It is correct as used in Equations B-6 and B-7, but N^*_{Fo} is needed for the following calculations.

If a Δp value could be found for a core plug such that N^*_{Fo} were equal to 0.005 (i.e., the value inside the brackets in Equation A-3 equals 1.005), then Darcy's equation (or equivalently Equation A-3 with a value of 1.000 inside the brackets) would underestimate k_∞ by only 0.5 percent. We will find this Δp by first setting N^*_{Fo} in Equation A-3 to 0.005, then solving for v_m:

$$v_m = \frac{C_1 k_\infty \left(1 + \frac{b}{P_m}\right)\Delta p}{C_2 \mu L (1.005)} \qquad \text{(A-5)}$$

This and the chosen value of N^*_{Fo} are substituted into Equation A-4, which is solved for Δp, keeping in mind that $P_m = P_2 + \frac{1}{2}\Delta p$:

$$\Delta p_{max} = (P_2 + b)\left(\sqrt{1 + \frac{2C_2^2 RT\mu^2 L(0.005)(1.005)}{C_1 C_3 M k_\infty k_g \beta (P_2 + b)^2}} - 1\right) \qquad \text{(A-6)}$$

Now the task remains to find appropriate values of β (in view of the large amount of scatter in a β vs. k_∞ plot). A slightly conservative approach was taken: values of β lying on the upper dashed curve in Figure 6-3 were divided by 10. This corresponds to a curve that lies above the central solid curve by about a factor of 3 for low values of k_∞, and gradually falls below the central curve at the highest permeabilities. Thus actual β's for the majority of core plugs would be expected to be somewhat less than the values chosen, and the error incurred from calculating permeability using the Darcy equation (provided that Δp_{max} as calculated from Equation A-6 is not exceeded during the permeability measurement) would be less than 0.5 percent. On the other hand, if a plug's actual β happened to lie on the upper dashed line, use of the same Δp_{max} would result in a calculated permeability that is 5 percent low. If the plug's actual β were twice the value shown by the upper dashed curve, the error would be 10 percent, etc.

The calculation of Δp_{max} is not completely straightforward. Equation A-6 contains b, k_g, and k_∞. For purposes of preparing Figure 6-4, b was found from a correlation proposed by Jones[16] for data on a large number of core plugs using helium:

$$b_{He} = 16.4 k_\infty^{-.0382} \qquad \text{(A-7)}$$

where b_{He} is the Klinkenberg gas slippage factor for helium, psi, and k_∞ is the slip-corrected permeability, millidarcys. To calculate b_{air}, the constant 16.4 in Equation A-7 becomes 5.71, using the technique illustrated by Equation 31. Values for C_1, C_2, C_3, and R for desired units are found from Table 6-1.

The required k_{air} is calculated from:

$$k_{air} = k_\infty \left(1 + \frac{b_{air}}{P_2 + \frac{1}{2}\Delta p_{max}}\right) \qquad \text{(A-8)}$$

Because Δp_{max} is unknown, use k_∞ as the first guess for k_g in Equation A-6, and solve for Δp_{max}. Insert this value into Equation A-8 to solve for k_{air}. Now use this value in Equation A-6 for k_g, and iteratively continue to refine the estimates until there is no further change in Δp_{max}. Convergence is usually achieved in 4 to 10 iterations.

Results of these calculations are shown in Figure 6-4 for downstream pressures of 14.7, 50, 100, and 200 psia for air at room temperature. The $\Delta p / L$ values are reported as "psi per inch of core plug length." Although these ratios are slightly dependent upon length, and were calculated for two-inch long plugs, results are close enough for the intended purpose for plugs ranging in length from 1 to 3 inches.

Starting with the highest permeabilities for the 14.7 psia downstream pressure, the maximum allowable pressure drop increases with decreasing permeability, as expected. However, for permeabilities below about 0.3 millidarcys, $\Delta p_{max} / L$ decreases with decreasing permeability. This unexpected result is a consequence of gas slippage. Slippage is greatest at the lowest permeabilities, causing higher gas flow rates than would occur without slippage. It is decreased when the mean pore pressure is increased at the higher back pressures.

At the higher permeabilities, $\Delta p_{max} / L$ values are reduced with increasing back pressure. This is mainly a consequence of increased gas density, which increases mass flux, and consequently inertial resistance, for a given velocity.

A.6.8.1.2 Liquids

Liquids are nearly incompressible and are not subject to slippage. Therefore the derivation for the maximum pressure drop to maintain the condition of Stokes flow is simpler than that for gases. Again, choosing a value of 0.005 for the Forchheimer number and the same beta vs. permeability relationship as for gases, the equation for liquids is:

$$\frac{\Delta p_{max}\rho_L}{L\mu_L^2} = \frac{5.764\text{E}12}{\beta k_L^2} \quad \text{(A-9)}$$

Where:

Δp_{max} = maximum pressure drop for Stokes flow, psi.
L = length of core plug, inches.
μ_L = viscosity of liquid, cp.
ρ_L = density of liquid, g/cm^3.
β = coefficient of inertial resistivity, ft^{-1}.
k_L = permeability of plug to liquid, millidarcys.

For a given β and k_L the left hand side of Equation A-9 applies for a core of any length. Results are plotted in Figure A.6-1. Use of this figure is illustrated by an example: Suppose we determined permeability of a 2.50 inch long core plug with a pressure drop of 35.8 psi and a liquid with viscosity of 1.53 cp. and density of 0.816 g/cm^3. A permeability of 99.8 millidarcys was calculated using Darcy's law. Is it likely that Darcy's law was valid to use?

From Figure A.6-1, a permeability of 99.8 yields a value of about 6.36, which is equal to the left hand side of Equation A-9:

$$\frac{\Delta p_{max}\rho_L}{L\mu^2} = 6.36$$

therefore:

$$\Delta p_{max} = \frac{6.36 \times 2.50 \times 1.53^2}{0.816} = 45.6 \text{ psi}$$

Because the Δp actually used for the measurement, 35.8 psi, was less than the maximum allowable value, we conclude that Darcy's equation probably gave valid results.

B.6.8.2 Calculation of Permeability From Pressure-Falloff, Axial Gas Flow Measurements

The equation for axial flow, used with the pressure falloff method proposed by Jones,[21] starts with the steady-state Forchheimer equation for isothermal axial flow, corrected for gas slippage. It is obtained by integration of Equation 17 (see 6.2.1.2), which yields:

$$\frac{(P_1 - P_2)(P_m + f_{Fo}b)z_r}{P_r q_r z_m} = A_1 + A_2\left(\frac{P_r q_r (P_m + b)}{z_r P_m}\right) \quad \text{(B-1)}$$

Where:

$$A_1 = \frac{C_2\mu L}{C_1 A k_\infty} \quad \text{(B-2)}$$

$$A_2 = \frac{C_3\beta M L}{C_1 A^2 R T} \quad \text{(B-3)}$$

$$A = \frac{\pi D^2}{4} \quad \text{(B-4)}$$

$$N_{Fo} = \frac{q_r P_r A_2}{z_r A_1} \quad \text{(B-5)}$$

$$E = \frac{bN_{Fo}}{1 + N_{Fo}} \quad \text{(B-6)}$$

and

$$f_{Fo} = \frac{\left(1 - \frac{E}{P_1 - P_2}\ln\left[\frac{P_1 + E}{P_2 + E}\right]\right)\left(\frac{P_1 + P_2 + 2E}{P_1 + P_2}\right) + N_{Fo}}{(1 + N_{Fo})} \quad \text{(B-7)}$$

The Forchheimer number, N_{Fo} (Equation B-5), which is the dimensionless ratio of inertial to viscous resistivities, is a "Reynolds number for flow through media." It is discussed more fully in Appendix A. The "Forchheimer interaction factor," f_{Fo} (Equation B-7), is a dimensionless number that has a maximum value of 1.0 (whenever b or N_{Fo} goes to zero). Its minimum value for any practical case is about 0.95.

The instantaneous gas flow rate entering the sample is determined from the total connected reservoir and manifold volume upstream of the inlet core face, V_T, and the pressure-time derivative:

$$q_{1,t} = \frac{V_T f_{z,t}}{P_{1,t}}\left(\frac{-dP_{1,t}}{dt}\right) = V_T f_{z,t}\left(\frac{-d\ln[P_{1,t}]}{dt}\right) \quad \text{(B-8)}$$

Where:

$$f_{z,t} = 1 - \frac{P_{1,t}}{z_{1,t}}\left(\frac{dz_{1,t}}{dP_{1,t}}\right) \quad \text{(B-9)}$$

is a correction for non-ideal gases. The subscript 1 in these equations refers to conditions just upstream of the inlet face of the sample. Because all flow rates, pressures (except atmospheric pressure), and gas deviation factors refer to this location, this subscript will be dropped in equations below. Subscripts n will refer to time, not position.

In general, it is more useful to measure a gauge pressure than the absolute pressure required by Equation B-8. Because the sample is vented directly to atmospheric pressure, the gauge pressure is equal to the Δp across the sample at any instant. If we multiply and divide Equation B-8 by p and realize that $dp = dP$, we obtain:

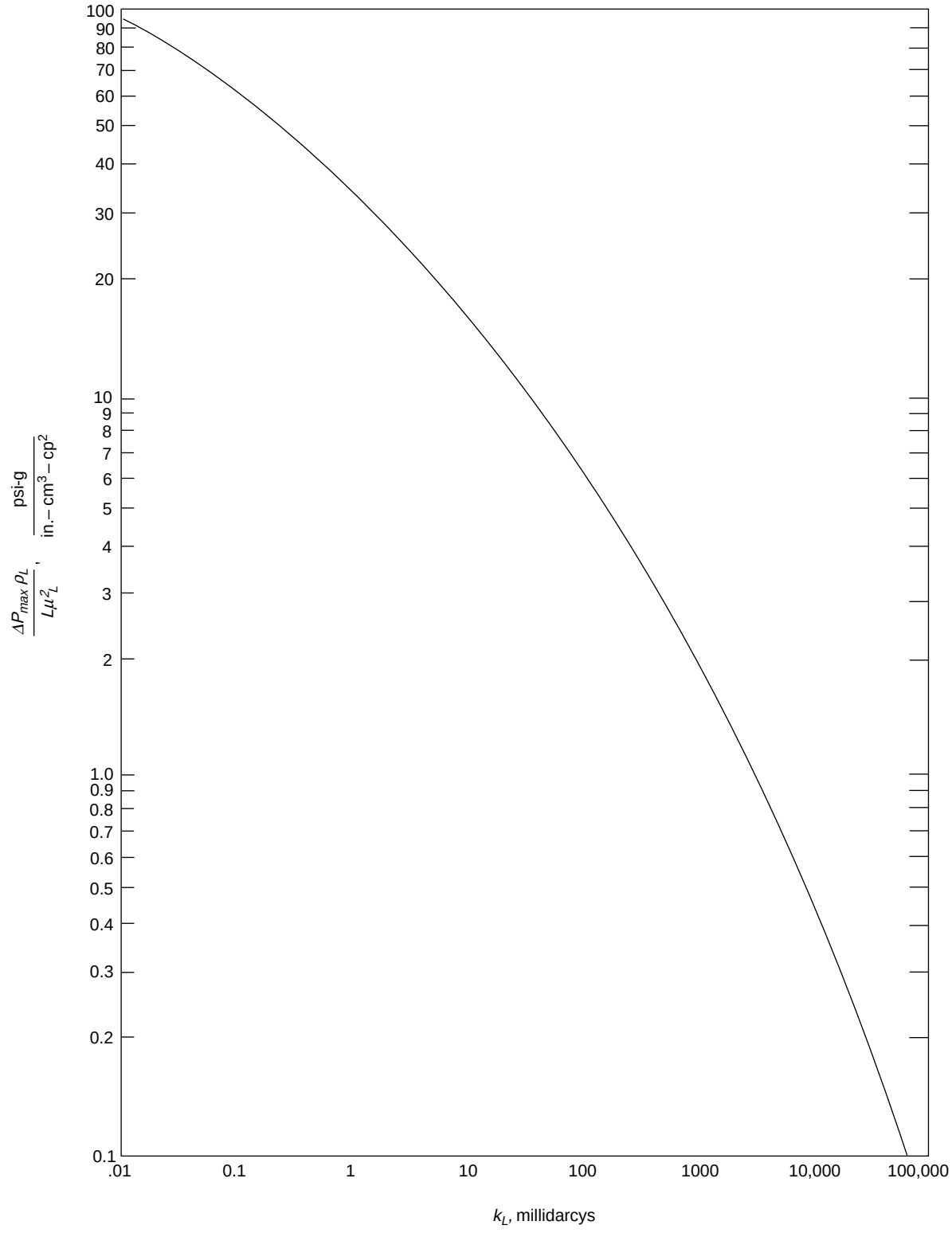

Figure A.6-1—Plot for Determining Maximum Allowable Δp in Axial Flow Liquid Permeability Measurements

$$q = \frac{-pV_T f_z}{pP}\left(\frac{dp}{dt}\right) = \frac{-pV_T f_z}{P}\left(\frac{d\ln p}{dt}\right) \qquad \text{(B-10)}$$

We can now define an instantaneous flow rate function, y:

$$y \equiv -V_T\left(\frac{d\ln p}{dt}\right) = \frac{qP}{f_z p} \qquad \text{(B-11)}$$

The derivative in Equation B-11 is accurately approximated from any pair of adjacent pressure-time points:

$$y_n = \frac{V_t \ln\left[\frac{p_{n-1}}{p_n}\right]}{t_n - t_{n-1}} \qquad \text{(B-12)}$$

This approximation most closely equals the true slope (derivative) of the ln[p] vs. time curve (see Figure B.6-1) at the pressure-midpoint of the interval, which is the geometric mean of the two pressures:

$$p_{g_n} = \sqrt{p_{n-1}p_n} \qquad \text{(B-13)}$$

Thus y_n is calculated for the instant when the upstream pressure is p_{g_n}. The mean pore pressure in the sample at this time is:

$$P_{m_n} = \tfrac{1}{2}p_{g_n} + P_a \qquad \text{(B-14)}$$

Now, except for one complication, any of the several sets of y_n, p_{g_n}, P_{m_n}, and f_{z_n} values obtained from the pressure falloff could be used to calculate the flow variables in Equation B-1. With steady-state flow, where the pressure at any point in the sample is invariant with time, the mass flow rate is constant throughout the length of the sample. The complication is that this is not true for unsteady-state flow.

Whenever pressure at any point in the sample decreases with time, gas density there decreases proportionally, with a resultant mass-depletion of gas. Because the upstream pressure decreases continually during a pressure falloff, the pressure throughout the length of the sample also decreases, except at the outlet end, which always remains at atmospheric pressure. The rate of mass depletion of resident gas in the sample is accompanied by an equal increase in the mass rate of flowing gas. Therefore, at any given time during a transient pressure decline, the mass flow rate of gas leaving a sample is

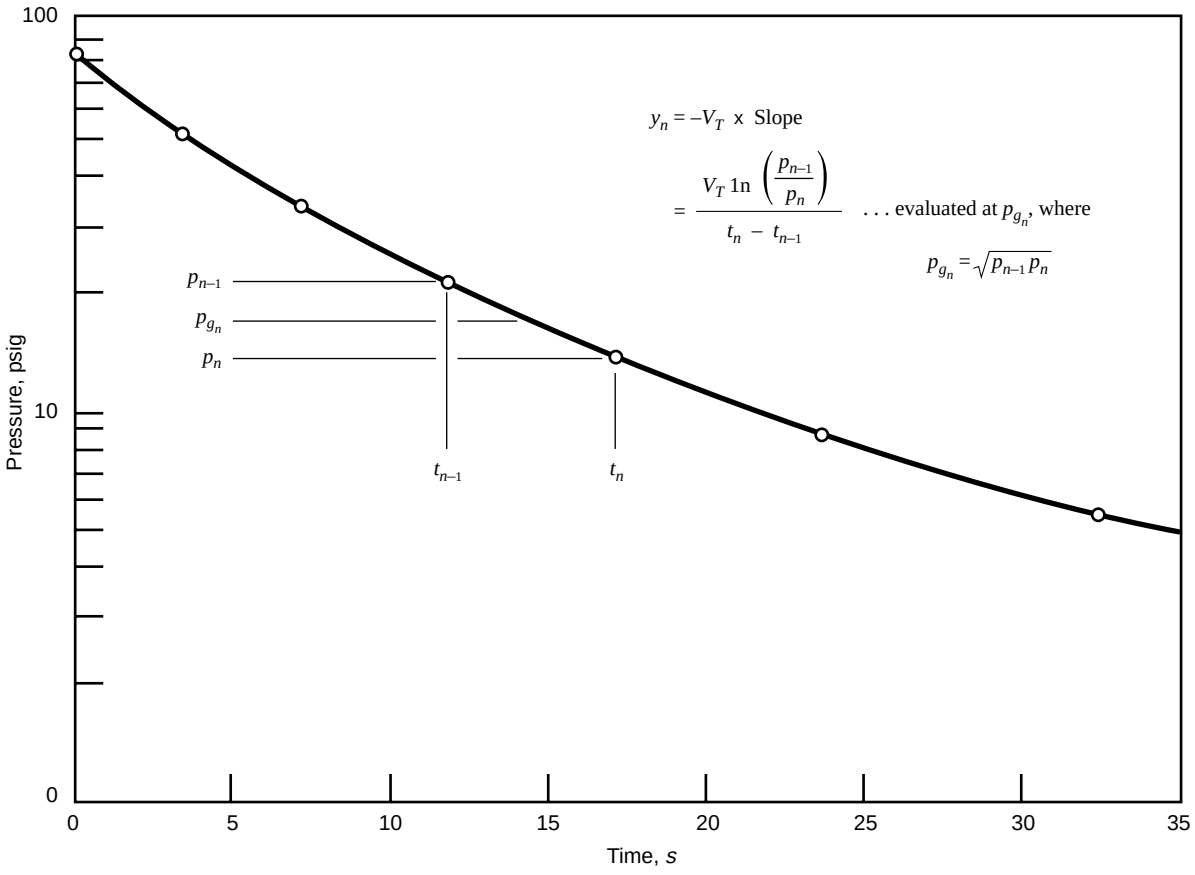

Figure B.6-1—Construction of Flow Variables y_n and p_{gn}

greater than the mass rate entering the sample. If the gas reservoir volume, V_T is large compared to the pore volume of the sample, V_p, then the relative increase is small. If these volumes are comparable, the increase is significant. In the limit, an infinite reservoir volume yields steady-state flow—no increase. At the opposite extreme, when the reservoir volume is zero, all the gas exiting from the sample originates from within the sample itself.

Because of the non-constant mass flux throughout the length of a sample at any instant during the pressure transient, we cannot use the steady-state solution directly. The correct solution can be achieved, however, by an iterative technique in which the continuity equation is decoupled from the flow equation, but where both equations are ultimately satisfied within an acceptable tolerance: the partial derivative of gas density with respect to time at any length, taken from the steady-state solution of the flow equation, is inserted into the continuity equation from which the approximate mass flux variation with length is derived for several conditions of pressure, slip-factor magnitude, and ratios of pore volume to reservoir volume. The corrected, non-constant mass flux is inserted into the differential form of the flow equation, which is then re-integrated with respect to length. The process is repeated until any further change is negligible. Four iterations were adequate for all practical cases. The corrected flow rate that can be inserted into the integrated flow equation to allow for the weighted average increase in mass flow with length at any instant is:

$$q_{c_n} = q_n(1 + \gamma G_m[c_n, \gamma]) \tag{B-15}$$

Where:

$$\gamma = \frac{2V_p}{3V_T} \tag{B-16}$$

and

$$c_n = \frac{(P_a + b)^2}{2p_{g_n}(P_{m_n} + b)} \tag{B-17}$$

The correction factors for non-constant gas mass flow, $G_m[c_n, \gamma]$, are shown as a function of c for several values of γ in Figure B6-2. We can now define a "corrected instantaneous flow rate function," γ_{c_n}:

$$y_{c_n} \equiv \frac{y_n f_{z_n}}{z_n}(1 + \gamma G_m[c_n, \gamma]) = \left(\frac{q_r P_r}{z_r p_g}\right) \tag{B-18}$$

where f_{z_n} (Equation B-9) is evaluated at pressure P_n, where $P_n = p_{g_n} + P_a$. This function can be inserted into Equation B-1 to produce the integrated form of the slip-corrected Forchheimer equation for transient flow:

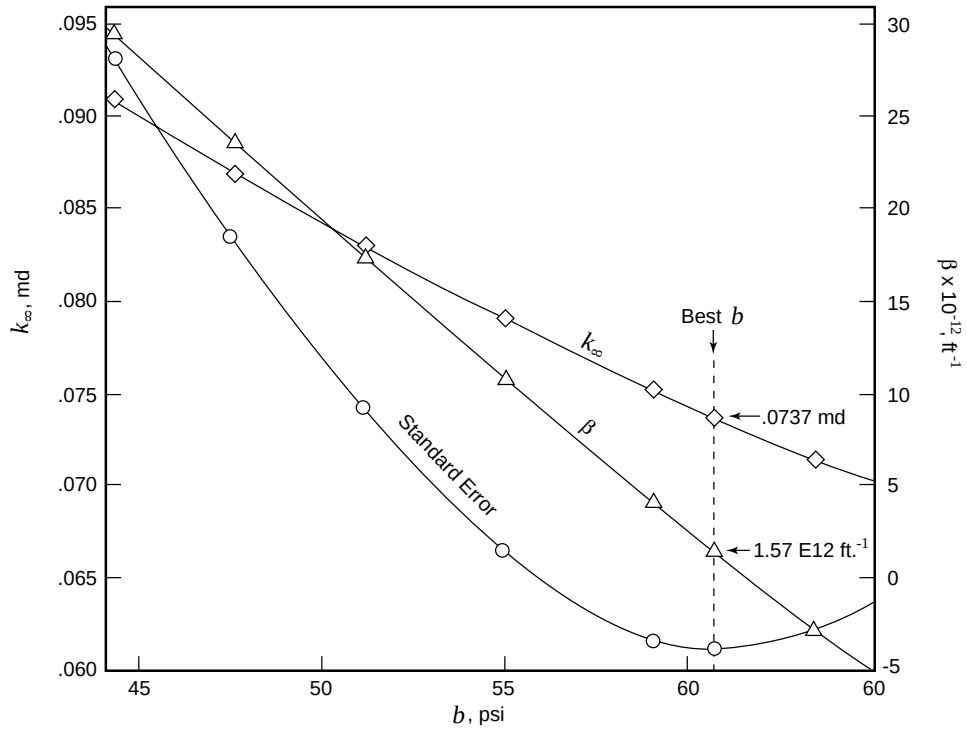

Figure B.6-2—Dimensionless Correction Factors for Non-Constant Mass Flux in Pressure-Falloff Permeability Measurements

$$\left(\frac{P_m + f_{Fo}b}{y_c z_m}\right)_n = A_1 + A_2\left(\frac{y_c p_g (P_m + b)}{P_m}\right)_n \qquad \text{(B-19)}$$

The Forchheimer number (Equation B-5) must be calculated for each f_{Fo_n} in the equation above. In terms of the corrected flow function, this is:

$$N_{Fo_n} = \frac{y_{c_n} p_{g_n} A_2}{A_1} \qquad \text{(B-20)}$$

E_n (Equation B-6) must also be calculated for each data point obtained in order to calculate f_{Fo_n} (Equation B-7).

The coefficients A_1 and A_2 in Equation B-19, which yield k_∞ and β, respectively, are determined from the set of equations corresponding to each data point, n, using linear regression as follows:

1. Calculate an initial guess for the slip factor, *b*. For helium, this is:

$$b[\text{first guess}] = 0.803\left(\frac{D^2}{\mu_{He} L y_{c_m}}\right)^{0.467} \qquad \text{(B-21)}$$

where y_{c_m} is the flow function that corresponds to the lowest p_g value obtained.
2. Set f_{Fo} for all *n*.
3. Calculate c_n (Equation B-17) and y_{c_n} (Equations B-12 and B-18) for each *n*.
4. Calculate the left-hand and right-hand terms in Equation B-19 for each *n*.
5. Perform a linear regression to obtain A_1 and A_2.
6. From these values, calculate N_{Fo} (Equation B-20), *E* (Equation B-6), and f_{Fo} (Equation B-7) for each *n*.
7. Repeat steps 4 through 6 until the change in any f_{Fo} does not exceed 0.001 from its value in the previous iteration. Three iterations are usually adequate.
8. Calculate a Standard Error, SE, from the last linear regression.
9. Choose a new *b* that is 10 percent higher than the first-guessed *b*.
10. Using the most recently calculated set of values for f_{Fo} and the new *b*, repeat steps 3 through 8.
11. If the new SE is lower than the previous one, increase *b* by 10 percent. Otherwise, reduce the original *b* by 10 percent. Repeat step 10.
12. Continue to increase or decrease *b*, repeating step 10, until the minimum SE is found. This corresponds to the best values of *b*, A_1 and A_2, in a least-squares sense. Calculate k_∞ and β from A_1 and A_2 (Equations B-2 and B-3), respectively.

Typical changes in k_∞, β, and SE with *b* are illustrated by Figure B.6-3. (The scale for the SE has been omitted for clarity.) As *b* increases, calculated values of both k_∞ and β decrease. The correct *b* for this example, which occurs at the minimum SE, is 60.6 psi. The calculated value of β becomes

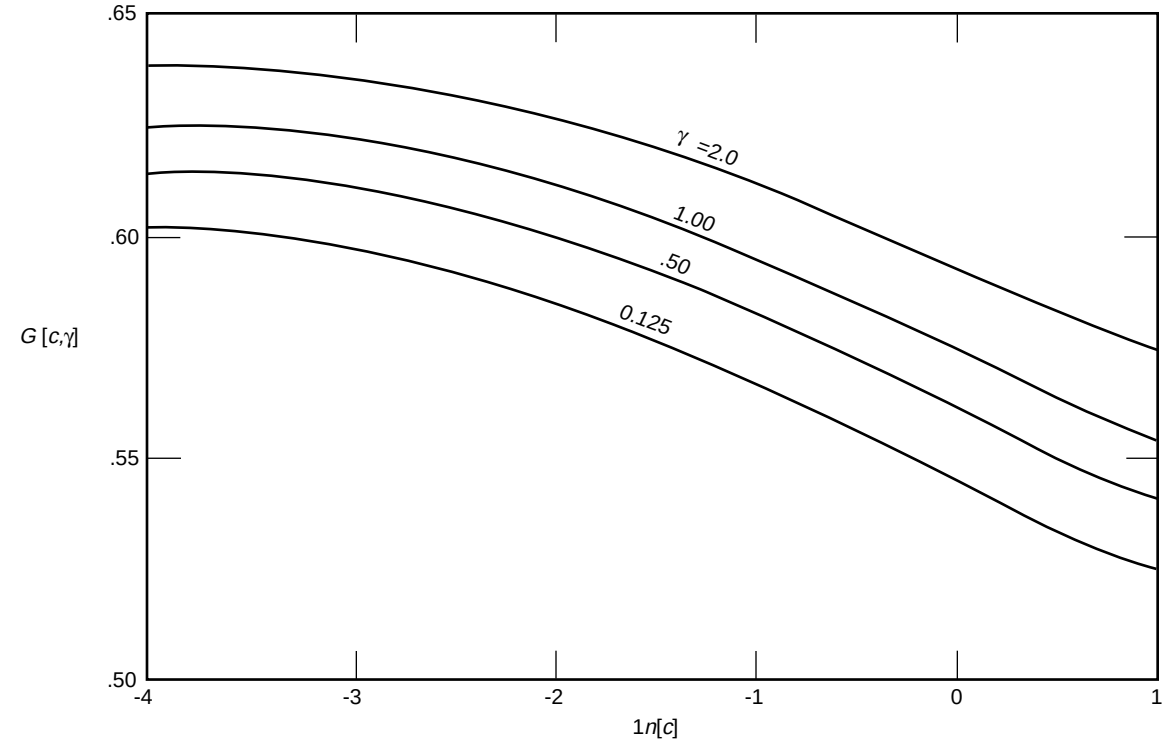

Figure B.6-3—Variation of k_∞, ß, and Standard Error with *b* for Pressure-Falloff Data

zero at a slip factor of 61.5 psi, and is negative for larger *b*s. A negative calculated β usually indicates that b is too high. However, extreme sensitivity of permeability to small changes in net stress can also cause negative βs. Changes in k_∞ and β with b, as shown in Figure B.6-3, are general, in the sense that they always decrease with increases in b.

The computational effort for the pressure falloff technique is considerably greater than for steady-state calculations. However, the entire procedure outlined above, with 25 y and p_g values each, is accomplished nearly instantaneously with a small computer.

Low-permeability rocks at small pressure differentials often exhibit little inertial resistance. In these cases, the term in Equation B-19 that is multiplied by A_2 is small, f_{Fo} is nearly equal to 1, and Equation B-19 can be approximated by:

$$y_{c_n} z_{m_n} = \frac{1}{A_1}(P_{m_n} + b) \tag{B-22}$$

For the approximation to be valid, a plot of $y_{c_n} z_{m_n}$ vs. P_{m_n} must be linear. Equation B-22 is the Darcy equation, corrected for gas slippage, for pressure-falloff measurements. Its slope and intercept are equal to $1 / A_1$ and b / A_1, respectively. Therefore k_∞ and b are calculated from:

$$k_\infty = \frac{C_2 \mu L(\text{slope})}{C_1 A} \tag{B-23}$$

and

$$b = \frac{(\text{intercept})}{(\text{slope})} \tag{B-24}$$

Departure from linearity, especially at the higher P_{m_n} values, indicates that inertial resistance is not negligible at these conditions.

C.6.8.3 Calculation of Permeability From Pressure-Falloff, Probe Gas Flow Measurements

Instantaneous flow rate functions, y_n, and geometric mean pressures, p_{g_n}, are calculated from Equations B-12 and B-13, respectively. These are inserted into:

$$\frac{\frac{1}{2}p_{g_n} + P_a + b^*}{y_n} = A_3 + A_4\left(\frac{y_n p_{g_n}(\frac{1}{2}p_{g_n} + P_a + b^*)}{\frac{1}{2}p_{g_n} + P_a}\right) \tag{C-1}$$

Where:

$$A_3 = \frac{C_2 \mu}{C_1 (G_0 r_i) k_\infty} \tag{C-2}$$

and

$$A_4 = \frac{C_3 \beta M}{C_1 G_{f_{fo}} RT} \tag{C-3}$$

The dimensional geometric factor for the Forchheimer term above, $G_{f_{Fo}}$, must be determined by numerical modeling. If this geometric factor can be determined, then β could be calculated from Equation C-3. Regardless, use of Equation C-1 corrects for inertial resistance, whether or not β can be calculated.

Note that except for the different geometrical factors contained in A_3 and A_4, Equation C-1 is virtually identical to Equation B-19 (for axial flow), except for certain simplifications in the probe-permeameter equations. These simplifications are: (a) gas deviation (z) and Forchheimer interaction (f_{Fo}) factors are omitted. Pressures are so low that these factors are nearly unity, and (b) y_n values are not corrected for non-constant (spatial) mass flow. With commonly used probe-seal dimensions, the rock pore volume that influences permeability measurement is so small, relative to the volume of even the smallest gas reservoir, that the steady-state assumption of constant mass flow through any isopotential surface at any instant does not introduce any appreciable error in the calculation of permeability. For example, the error with an interior probe seal radius of 3.8 millimeters, and reservoir volume of 5.2 cm^3 is less than 0.1 percent.

The b^* that appears in Equation C-1 is a gas slippage factor taken from a correlation. Although b could theoretically be determined directly from the pressure falloff data, the range of mean pore pressures measured (with a fill pressure of 10 psig or less) is too small to determine b reliably. The correlation b^* is obtained using an iterative technique. Its first approximation (for nitrogen, in psi) is calculated from:

$$b^*_{N_2} = 0.22\sqrt{\frac{(G_0 r_i)}{\mu_{N_2} y_m}} \tag{C-4}$$

where y_m corresponds to the lowest value of p_g obtained from the pressure-falloff data. This first-guess value of b^* is used in Equation C-1 to calculate its left and right hand terms for each y_n and p_{g_n} value obtained. Next, the coefficients A_3 and A_4 are found from Equation C-1 using linear regression. The first estimate of the Klinkenberg permeability is obtained from rearrangement of Equation C-2:

$$k_\infty = \frac{C_2 \mu}{C_1 (G_0 r_i) A_3} \tag{C-5}$$

Using this k_∞, an improved estimate of $b^*_{N_2}$ is calculated from the correlation:

$$b^*_{N_2} = 6.9 k_\infty^{-0.382} \tag{C-6}$$

This new value of b^* is reinserted into Equation C-1, and the process is repeated until the change in b^*, from one iteration to the next, is less than 0.1 psi. Convergence should be obtained within 2 to 4 iterations.

Inertial resistance virtually disappears for samples with permeabilities of less than 0.1 millidarcy for starting pressures of less than 10 psig. For these samples, the A_4 term in Equation C-1 can be ignored, and the Forchheimer equation reduces to the slip-corrected Darcy equation:

$$k_\infty = \frac{C_2 \mu y^*}{C_1 (G_0 r_i)(\frac{1}{2} p_g^* + P_a + b^*)} \qquad \text{(C-7)}$$

This equation can be used when as few as one to three $y - p_g$ points are obtained for low permeability samples. The y^* and p_g^* refer to any one of these points. The calculation is again iterative. The initial estimate of b^* is calculated from Equation C-4. The estimate is refined by calculating k_∞ (Equation C-7), which is then used in Equation C-6. The process is continued until the change in b^*, from one iteration to the next, is less than 0.1 psi. Convergence is usually achieved in 2 to 4 iterations.

Gas permeability, k_g, for any desired gas at any mean pore pressure can be calculated from k_∞ and b^* as outlined in 6.4.1.1.5 for the axial flow case.

D.6.8.4 Calculation of Permeability From Pulse Decay, Axial Gas Flow Measurements

The initial pressure difference across the sample, $\Delta p[0]$, which is slightly less than Δp_1 (see 6.4.1.3.2), must be calculated from a gas mass balance:

$$\Delta p[0] = \Delta p_1 \left(\frac{V_1 - V_d}{V_1} \right) \qquad \text{(D-1)}$$

V_1, the total upstream volume, includes: internal volumes of the upstream reservoir; the upstream chamber of the differential pressure transducer; connecting lines to the transducer and valves 1, 2, and the fill valve; and the upstream dead volume, V_d, (the volume inside valve 1); the upstream end plug, and the line connecting them. The total downstream volume, V_2, includes the volumes in the downstream reservoir, transducer, and end plug; the downstream chamber of the differential transducer; and the lines connecting them, including the line to valve 2. The volumes V_1, V_2, and V_d must all be found from careful calibration using Boyle's Law techniques (see 6.6.3.1).

The differential pressure and downstream pressure are monitored as functions of time. Data are analyzed from the solution of the diffusivity equation, that is derived by combining the differential form of Darcy's Law with the continuity equation. The sample is presumed to be initially at a uniform pore pressure throughout. Then at time $t = 0$, a pulse of slightly higher pressure, $\Delta p[0]$, is applied to its upstream end from the upstream reservoir. As gas flows from V_1 into the sample, the pressure in V_1 declines. The pressure in V_2 remains constant for a short period of time until the pressure pulse has traversed the length of the sample. Then the pressure in V_2 rises. Because $P_1[t]$ declines and $P_2[t]$ rises, $\Delta p[t]$ continues to diminish and gradually approaches zero as the upstream and downstream pressures become equal. The rate of pressure decay depends on permeability: the lower the permeability the slower the decay. The general solution for the pressure difference as a function of time as presented by Dicker and Smits,[23] following the original work of Brace et al.,[24] the error-function solution of Bourbie and Walls,[25] the general analytical solution of Hsieh et al.,[26] Chen and Stagg,[27] Haskett et al.,[28] and others is:

$$\frac{\Delta(P[t])^2}{\Delta(P[0])^2} = 2\sum_{m=1}^{\infty} \frac{a(b^2 + \theta_m^2) - (-1)^m b\sqrt{(a^2 + \theta_m^2)(b^2 + \theta_m^2)}}{\theta_m^2(\theta_m^2 + a + a^2 + b + b^2) + ab(a + b + ab)} \cdot \exp[-\theta_m^2 t_D] \qquad \text{(D-2)}$$

Where θ_m are roots of the equation:

$$\tan\theta = \frac{(a+b)\theta}{\theta^2 - ab} \qquad \text{(D-3)}$$

a, the ratio of the compressive storage (the product of volume and compressibility) of the sample's pore volume to that of the upstream reservoir, is:

$$a = \frac{V_P(c_g + c_{pv})}{V_1(c_g + c_{v_1})} \qquad \text{(D-4)}$$

b, the ratio of the compressive storage of the sample's pore volume to that of the downstream reservoir, is:

$$b = \frac{V_P(c_g + c_{pv})}{V_2(c_g + c_{v_2})} \qquad \text{(D-5)}$$

and t_D, dimensionless time, is:

$$t_D = \frac{C_1 k_g t}{C_2 \mu \phi (c_g + c_{pv}) L^2} \qquad \text{(D-6)}$$

where C_1 and C_2 are conversion constants, given in Table 6-1, required to make t_D dimensionless. The c_g, c_{pv}, c_{v_1}, and c_{v_2} are compressibilities of gas, the sample's pore volume, and upstream and downstream reservoirs, respectively. Gas compressibility is:

$$c_g = \frac{1}{P}\left(1 - \frac{d\ln[z]}{d\ln[P]}\right) \equiv \frac{f_z}{P} \qquad \text{(D-7)}$$

In Equation D-7, we have defined f_z as the value of the group within parentheses that accounts for deviation from ideal gas behavior. At pressures of interest, this deviation is not negligible. Values of f_z for nitrogen at 72°F are given by Jones.[44] If the system is built rigidly, i.e., with thick-walled vessels, and if displacement volumes of the transducers are

small relative to V_1 and V_2, then c_{V_1} and c_{V_2} are small compared to c_g, and can be ignored. Pore-volume compressibility varies considerably among samples of different lithologies. However, it is minimized by using high net confining stresses, and is usually less than 2×10^{-5} psi^{-1} for net stresses of 5,000 psi or more. A c_{pv} of 1×10^{-5} psi^{-1} is equal to 1 percent of the gas compressibility at a gas pressure of 1000 psia, or 2 percent at 2,000 psia.

The squared pressure differences (that apply to gases only) that appear on the left hand side of Equation D-2 are:

$$\frac{\Delta(P[t])^2}{\Delta(P[0])^2} \equiv \frac{P_1[t]^2 - P_2[t]^2}{P_1[0]^2 - P_2[0]^2} = \frac{\Delta p[t](P_2[t] + \frac{1}{2}\Delta p[t])}{\Delta p[0](P_2[0] + \frac{1}{2}\Delta p[0])} \quad \text{(D-8)}$$

Equation D-2 applies for all values of a, b, and t_D, provided that all roots of Equation D-3 are used, and all compressibilities are constant. Haskett et al.[28] minimize the effect of the restriction by incorporating time-varying compressibility into an "adjusted pseudo time." However, with the proper experimental conditions, neither of the restrictions poses a serious problem, as will be discussed below. The use of the squared pressure differences in Equation D-2 is nearly equivalent to using pseudo-pressures as suggested by Haskett et al. Because the change in the mean pore pressure is so small in the technique outlined here, changes in the *z*-factor and gas viscosity are small. Their ratios between the numerator and denominator of Equation D-9 are virtually unity. For experimental conditions other than outlined below, where variations in the mean pore pressure are greater, the use of pseudo-pressures is recommended.

The choice of reservoir sizes is not limited directly by Equation D-2. Some investigators have used large upstream vessels ($a \cong 0$) or large downstream vessels ($b \cong 0$). Others have advocated reservoir volumes that are similar in magnitude to the pore volume of the sample (a or $b \cong 0.2$ to 5). In general, to keep measurement times reasonably short with low-permeability samples, at least one of the reservoirs must be small.

Dicker and Smits[23] present compelling reasons to make the upstream and downstream volumes identical and fairly small. The equal volumes create a desirable situation of symmetry. Following the period when the pressure pulse traverses the length of the sample, the pressure decrease in the upstream vessel is offset by an equal downstream pressure increase, thereby keeping the mean pore pressure constant and reducing the overall pore volume compressional changes nearly to zero. Because the average pressure in the upstream half (of a homogeneous sample) decreases with time, while the average pressure in the downstream half increases by the same amount, the mass flow rate of gas entering the sample is equal to that leaving the sample at the same instant. The maximum rate at any instant occurs about midway along the length of a reasonably homogeneous sample.

Another consequence of symmetry (where $a = b$) is that all even roots of Equation D-3 cancel out in Equation D-2. The first root of Equation D-3, θ_1, is slightly less than the square root of $(a + b)$ for small values of a and b (large reservoirs). If we define f_1 as:

$$f_1 \equiv \frac{\theta_1^2}{a + b} \quad \text{(D-9)}$$

then $f_1 = 1.000$ when $a + b = 0$, and gradually decreases as $a + b$ becomes larger, as illustrated in Figure D6-1. In this figure, V_s is the smaller of V_1 or V_2, and V_L is the larger of the two reservoir volumes.

The square of the first root of Equation D-3 is found from:

$$\theta_1^2 = f_1(a + b) \quad \text{(D-10)}$$

The higher roots of Equation D-3 are equal to:

$$\theta_m = (m - 1)\pi, \text{ for } (a + b) = 0 \quad \text{(D-11)}$$

and increase gradually as $(a + b)$ increases.

Except for early times when t_D is small, only the first term in the summation (Equation D-2) is significant. The second term is zero when a equals b. At a t_D of 0.1, all higher terms add only 0.16 percent to the contribution of the first term when $a = b = 1$, and less for smaller values of a and b. Therefore, if early times are not used, Equation D-2 reduces to a single exponential, which can be written as:

$$\ln\frac{\Delta(P[t])^2}{\Delta(P[0])^2} = \ln f_0 - \left(\frac{\theta_1^2 k_g}{(c_g - c_{pv})\mu\phi L^2}\right)t \quad \text{(D-12)}$$

Where:

$$f_0 = \frac{2[a(b^2 + \theta_1^2) + b\sqrt{(a^2 + \theta_1^2)(b^2 + \theta_1^2)}]}{\theta_1^2(\theta_1^2 + a + a^2 + b + b^2) + ab(a + b + ab)} \quad \text{(D-13)}$$

Thus, if experimentally obtained values of $\ln[\Delta(P[t])^2 / \Delta(P[0])^2]$ were plotted vs. time, a straight line would result (except for early times), which has an intercept of $\ln[f_0]$, and a negative slope of absolute value:

$$|slope| = \frac{C_1\theta_1^2 k_g}{C_2(c_g + c_{pv})\mu\phi L^2} = \frac{C_1 f_1(a + b)k_g}{C_2(c_g + c_{pv})\mu\phi L^2} \quad \text{(D-14)}$$

where C_1 and C_2 are constants from Table 6-1, used to make the units consistent. If c_{v_1} and c_{v_2} are negligible compared to c_g, then:

$$|slope| = \frac{C_1 f_1 A k_g P_m}{C_2 \mu L f_z}\left(\frac{1}{V_1} + \frac{1}{V_2}\right) \quad \text{(D-15)}$$

The mean pore pressure in Equation D-15, P_m, is an absolute pressure. When $a = b$, and the rock sample is homogeneous (or if a and b are small, e.g., less than 0.3), P_m should not change during the linear portion of the semi-log decay. It is calculated from:

$$P_m[t] = P_2[t] + \tfrac{1}{2}\Delta p[t] \qquad \text{(D-16)}$$

Calculation of the slope can be performed during the measurement, using the latest two time-pressure readings:

$$|slope| = \frac{\ln\left(\dfrac{\Delta p[t_{n-1}](P_2[t_{n-1}] + \frac{1}{2}\Delta p[t_{n-1}])}{\Delta p[t_n](P_2[t_n] + \frac{1}{2}\Delta p[t_n])}\right)}{(t_n - t_{n-1})} \qquad \text{(D-17)}$$

The absolute value of this slope will be high initially (if $a \cong b$), and will decrease fairly quickly, becoming constant with increasing time. After the slope has become constant for several readings, the intercept and slope of the linear portion of the decay can be obtained by linear regression of:

$$\ln\left(\frac{\Delta p[t_n]P_m[t_n]}{\Delta p[0]P_m[0]}\right) = A_0 + A_1 t_n \qquad \text{(D-18)}$$

where A_0 is the intercept and A_1 is the slope. Only data points yielding the constant slope (from Equation D-17) are to be used in the regression. From Equation D-12, we see that f_o can be obtained from the intercept:

$$f_0 = \exp[A_0] \qquad \text{(D-19)}$$

Equation D-13 shows that f_0 is a function of a and b, as is θ_1. Remarkably, when $a = b$, f_1 is only slightly greater than f_0: 0.0055 percent for $a = 0.1$; 0.12 percent for $a = 0.5$; and 0.44 percent for $a = 1.0$. Values of θ_1^2, f_1, and f_0 are listed in Table D.6-1 for various values of a, when $a = b$. These can be calculated from Equations D-3, D-10, and D-13 when the reservoirs are not of equal volume (more precisely, of equal compressive storage).

Figure D.6-2 shows f_1 as a function of f_0 for different ratios of V_2 / V_1. Because V_1 and V_2 are both determined by calibration, f_1 can be determined uniquely from f_0, and permeability calculated from:

$$k_g = \frac{-A_2 C_2 \mu L f_z}{C_1 f_1 A P_m\left(\dfrac{1}{V_1} + \dfrac{1}{V_2}\right)} \qquad \text{(D-20)}$$

Using the value of f_0 obtained, the pore volume of the sample can be estimated from:

$$V_p = \frac{aV_1}{1 + P_m c_{pv} / f_z} \qquad \text{(D-21)}$$

where a is found from Table 6-10, or from Figure D.6-1. If c_{pv} is estimated to equal 8×10^{-6} psi^{-1}, which may be too high or too low by a factor of 2 to 4 compared to the true value at a high net effective stress (4,000 to 5,000 psi), the maximum error from the assumption is about 1.6 percent for $P_m = 1000$ psia, or 3.2 percent for $P_m = 2{,}000$ psia. At lower net stresses, c_{pv} can be expected to be higher and more variable.

The differential pressure transducer should be electrically or mathematically zeroed just before valve 2 is closed (when the true differential pressure is zero at $P_2[0]$). If the transducer maintains its linearity, even though its gain or sensitivity may shift slightly in going from a low to a higher reference pressure (i.e., when P_2 is raised from atmospheric pressure to 1,000-2,000 psia), no error is incurred (except in the calculation of P_m) because all the Δp values are relative; they occur as ratios.

Table D.6-1—Pulse Decay Constants When $a = b$ ($V_1 = V_2$, or $S_1 = S_2$)

$a = b =$	θ_1^2	$f_1 = \theta_1^2/(a + b)$	$f_0 = \exp$[intercept]	f_1 / f_0
0.0	.000000	1.000000	1.000000	1.000000
0.1	.196711	.983553	.983500	1.000055
0.2	.387016	.967539	.967334	1.000212
0.3	.571166	.951944	.951500	1.000466
0.4	.749404	.936755	.935999	1.000808
0.5	.921963	.921963	.920826	1.001235
0.6	1.089064	.907553	.905980	1.001737
0.7	1.250923	.893516	.891458	1.002309
0.8	1.407744	.879840	.877255	1.002947
0.9	1.559725	.866514	.863366	1.003646
1.0	1.707053	.853527	.849789	1.004399
1.1	1.849910	.840868	.836516	1.005202
1.2	1.988469	.828529	.823544	1.006053
1.3	2.122896	.816499	.810866	1.006947
1.4	2.253350	.804768	.798477	1.007878

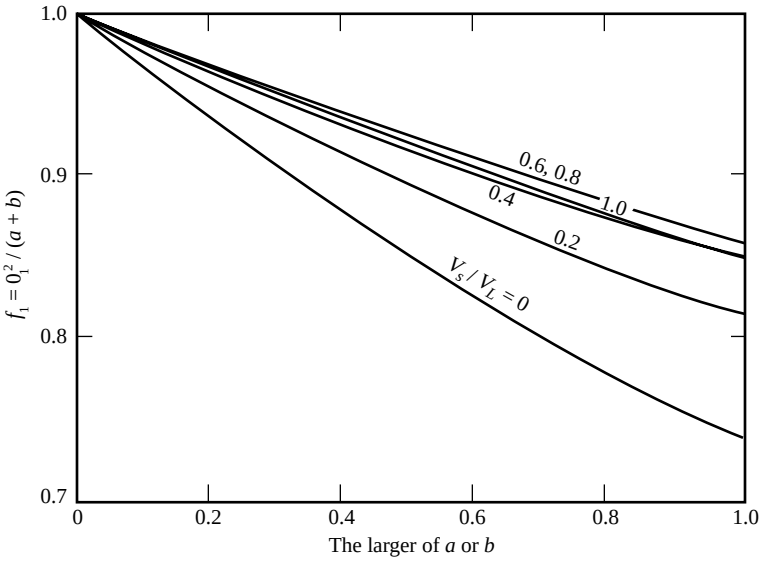

Figure D.6-1—First Root Relationship for Pulse-Decay Equation

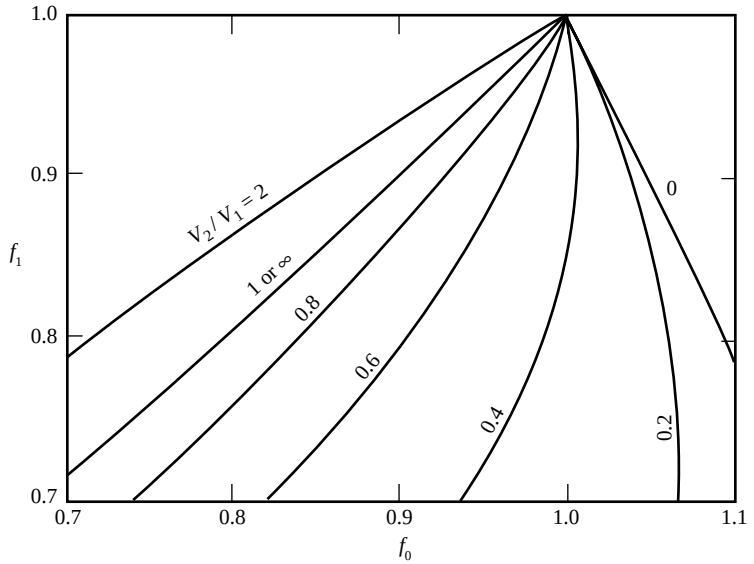

Figure D.6-2—Pulse-Decay Plot for Finding f_1, from f_0 with Known Ratios of V_2 / V_1 (for Gases) or S_2 / S_1 (for Liquids)

The practical range of permeability for gas pulse-decay measurements is about 0.1 millidarcy to 0.01 microdarcy (1 E-4 to 1 E-8 μm^2). The pulse-decay time is nearly proportional to sample length and inversely proportional to permeability and mean pore pressure. The time also depends strongly on the how much of the total pulse decay is measured.

As a general guideline, the reservoir volumes, V_1 and V_2, should be at least as large as the largest pore volume anticipated, and thereby 2-10 times the pore volume of the majority of samples to be run. The onset of the linear semi-log portion of the pulse decay occurs at about the same elapsed time regardless of the *PV*-reservoir volume ratio. The work of Kamath et al.[29] shows that the sensitivity to permeability heterogeneity is also reduced as *a* and *b* become smaller (larger reservoirs).

The upper permeability limit can be extended by increasing the reservoir volumes. However the sensitivity (thus the accuracy) of the pore volume determination (Equation D-21) is reduced with larger volumes.

Perhaps the most time-consuming part of this technique is allowing the system to reach true pressure equilibrium prior to generation of the pressure pulse. As pressures become uniform everywhere in the system, driving forces approach zero. If the pore volume of a sample is determined independently, the pressure equilibration step can be eliminated as suggested by Jones.[44]

E.6.8.5 Calculation of Permeability From Pulse Decay, Axial Liquid Flow Measurements

E.6.8.5.1 Apparatus and Measurement of Compressive Storage

One convenient method of measuring compressive storages in apparatus for liquid pulse decay measurements is to include a needle valve with a micrometer screw (as illustrated in Figure 6-25), which has a total calibrated displacement volume (cross-sectional area of the plunger at the stem seal multiplied by the length of its available stroke) of about 0.02 to 0.05 cm^3. Valves 1 and 2 should be of the type that have no internal volume change when opened or closed. To measure the storages, load a solid steel cylinder into the sample holder and apply confining stress. With valves 1 and 2 open, apply a vacuum to the fill/vacuum valve to remove all air from the apparatus. Then fill the entire system with degassed liquid to about 80 percent of the full scale pressure of the downstream pressure transducer. After the pressure has stabilized, close valves 1 and 2. The needle valve, valve 3, should be near fully-open. The differential pressure transducer should read zero, and the p_2 reading should not change when valves 1 and 2 are closed. Record the micrometer reading. Then screw in the micrometer, decreasing the upstream system volume until the differential pressure transducer reads full scale, *e.g.*, 100 psi. This pressure rise from the initial zero reading is Δp_1, and the volume decrease, calculated from the micrometer readings, is ΔV_1.

Because the volume between valve 1 and the upstream end of the steel plug in the sample holder (the "upstream dead volume") was excluded from the compression measurement, the "upstream compressive storage," S_1, less the "dead volume compressive storage," S_d, is calculated from:

$$S_1 - S_d = \frac{\Delta V_1}{\Delta p_1} \tag{E-1}$$

To determine S_d, open valve 1. After the differential pressure reading stabilizes to Δp_2, calculate the storage for the upstream dead volume from:

$$S_d = \Delta V_1 \left(\frac{1}{\Delta p_2} - \frac{1}{\Delta p_1} \right) \tag{E-2}$$

and the upstream compressive storage from:

$$S_1 = \frac{\Delta V_1}{\Delta p_2} \tag{E-3}$$

To determine the downstream storage, first unscrew the needle valve just past—then back to—the initial reading. As a check, the differential transducer should again read zero, and the downstream transducer should display its initial reading. Open valve 2. Read the downstream transducer pressure, $p_2[1]$. Screw in the needle valve until the stabilized downstream pressure, $p_2[2]$, is nearly equal to the full-scale reading of this transducer. Calculate the volume decrease, ΔV_2, from the micrometer readings, and the downstream compressive storage from:

$$S_2 = \frac{\Delta V_2}{p_2[2] - p_2[1]} - \frac{\Delta V_1}{\Delta p_1} - S_d \tag{E-4}$$

These calibrations of compressive storage must be performed for each different liquid used.

E.6.8.5.2 Procedure for Permeability Measurement

For a pulse decay liquid permeability measurement, a completely liquid-saturated rock sample is loaded into the sample holder, which is then pressurized, preferably to a fairly high (usually hydrostatic) confining stress. The system is filled with liquid, as described above, and pressurized to the initial pore pressure. The fill valve is left open long enough to allow the high pressure liquid to diffuse into the sample. With valves 1 and 2 still open, the fill valve is closed, and the downstream transducer is monitored until no further change in pressure is observed. Then valves 1 and 2 are closed and the pressure pulse is generated by screwing in the needle valve. After all pressures have stabilized, $S_1 - S_d$ is calculated from Equation E-1. This should have the same value as that obtained from the calibration.

The pulse decay is initiated by opening valve 1. The initial pressure difference across the sample, $\Delta p[0]$, will be slightly less than $\Delta p[1]$, which was generated with the needle valve with valve 1 closed, thereby excluding the upstream dead volume from the upstream volume. Therefore, $\Delta p[0]$ must be calculated, using the $\Delta p[1]$ and ΔV_1 that correspond to the run-time pressure-pulse generation, and the S_d that was determined from the calibration:

$$\Delta p[0] = \frac{1}{\frac{S_d}{\Delta V_1} - \frac{1}{\Delta p_1}} \tag{E-5}$$

E.6.8.5.3 Pulse Decay Calculations

Equations D-2 and D-3 apply to liquid, as well as gas, pulse decay measurements. However, the squared pressure differences on the left hand side of Equation D-2 must be replaced by first-power pressure differences for liquids:

$$\frac{\Delta p[t]}{\Delta p[0]} = 2\sum_{m=1}^{\infty} \frac{a(b^2 + \theta_m^2) - (-1)^m b\sqrt{(a^2 + \theta_m^2)(b^2 + \theta_m^2)}}{\theta_m^2(\theta_m^2 + a + a^2 + b + b^2) + ab(a + b + ab)} \cdot \exp[-\theta_m{}^2 t_d] \tag{E-6}$$

The a and b now refer to the ratios of liquid compressive storages:

$$a = \frac{V_p(c_L + c_{pv})}{V_1(c_L + c_{v_1})} \equiv \frac{S_{pv}}{S_1} \tag{E-7}$$

and

$$b = \frac{V_p(c_L + c_{pv})}{V_2(c_L + c_{v_2})} \equiv \frac{S_{pv}}{S_2} \tag{E-8}$$

and k_g and c_g in Equation D-6 are replaced by k_L and c_L, respectively.

After the early-time portion of the pressure decay, a plot of the natural logarithm of $\Delta p[t]/\Delta p[0]$ vs. time results in a straight line:

$$\ln\left[\frac{\Delta p[t_n]}{\Delta p[0]}\right] = \ln[f_0] - \left[\frac{C_1 f_1 kA}{C_2 \mu L}\left(\frac{1}{S_1} + \frac{1}{S_2}\right)\right] t_n \tag{E-9}$$

or, equivalently:

$$\ln\left[\frac{\Delta p[t_n]}{\Delta p[0]}\right] = A_0 + A_1 t_n \tag{E-10}$$

A_0 and A_1, which are the intercept and slope of the straight line, are found from Equation E-10 using linear regression on the linear portion only of the pressure decay data. The value of f_0, defined by Equation D-14, is calculated from the intercept:

$$f_0 = \exp[A_0] \tag{E-11}$$

The parameter f_1, which is needed for the calculation of permeability, is found from f_0 and the ratio of the calibrated downstream and upstream storages, S_2 / S_1, with the aid of Figure 6.D-2. Permeability is calculated from:

$$k_L = \frac{-A_1 C_2 \mu L}{C_1 f_1 A\left(\frac{1}{S_1} + \frac{1}{S_2}\right)} \tag{E-12}$$

Amaefule, et al.[30] used pore pressures ranging from 500 to 4,500 psi. Kamath, et al.[29] recommend pore pressures of nearly 1000 psi and pulse sizes of about 20 percent of the pore pressure, or more preferably, about 10 percent of the net confining stress. Volumes of the upstream and downstream reservoirs should be equal if possible, for reasons given in the gas pulse-decay section. They should be slightly smaller or about the same volume as the pore volume of the rock to be measured if heterogeneity is to be investigated, or 5 to 10 times larger, or more, if average permeabilities, comparable to steady-state values, are desired. See Kamath, et al.,[29] regarding “early time solutions” for investigation of heterogeneity, which is beyond the intended scope of this document.

SECTION 7—SUPPLEMENTARY TESTS

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7 Supplementary Tests

7.1 GENERAL

Acid solubility or calcimetry, oil gravity, core water salinity, grain size distribution, basic oil characterization, and petrographic characterization are supplementary data frequently obtained in conjunction with core analyses. Accordingly, these tests have been included because the data are useful in understanding and interpreting the core analysis results.

7.2 PETROGRAPHIC CHARACTERIZATION

Petrographic characterization is an important part of core analysis. Together, petrographic information and basic core analysis measurements provide an integrated framework for reservoir evaluations, including log interpretation. With geologic data, the results of core analyses can be extended to parts of a reservoir where there are no core measurements. Petrographic analysis also provides a basis for interpreting core analysis results, especially those that are unexpected or anomalous. Unexpected results may be valid and caused by variations in rock texture and/or mineralogy.

7.2.1 Petrographic characterization is best performed on the core plug used for routine core analysis so that the data are directly applicable. In those cases in which the plug cannot be consumed, the end sections obtained in preparing the core test samples or the core material from the same bedding plane as the test samples can be used. A minimum of one cubic inch (approximately 40 grams) of core should be saved for petrographic characterization.

7.2.2 Description of core analysis samples provides vital information on sample quality and character. Descriptions should be performed on extracted samples with the aid of a good binocular microscope. Visual porosity and pore type (e.g. intergranular, vuggy, moldic), drilling—or coring—induced features (e.g., drilling fluid invasion or induced fractures), and natural heterogeneity (clay laminae, lithologic variation normal to bedding, mineralized fractures, etc.) should be recorded. Petrographic characterization should include textural information and major mineralogical components. Textural data should include information on grain size, sorting, rounding, and grain shape. Mineralogical observations should include information on grain types and abundance. Approximate amounts of cement and matrix components can also be estimated with a binocular microscope in many cases.

7.2.3 Quantitative or semi-quantitative petrographic information can be obtained from thin sections, x-ray diffraction (XRD), bulk rock chemical analysis (usually by x-ray fluorescence, XRF), and Fourier transform infrared spectroscopy (FTIR). Valuable information regarding the spatial distribution of clay minerals within a sample can be obtained from scanning electron microscopy (SEM).

7.3 GRAIN SIZE DISTRIBUTION

Grain size distribution data have: (a) engineering application in well completion programs in friable and unconsolidated sediments, (b) geological application in assessing sand heterogeneity and depositional environment interpretation in both consolidated and unconsolidated clastic sediments, and (c) petrophysical application in various phases of formation evaluation to effect an understanding of log responses. Grain size distribution data are commonly utilized in sidewall core analysis to derive permeability. Methods commonly employed include wet and dry sieving, settling tube particle size analysis, thin section grain size analysis, and laser diffraction particle size analysis.

7.3.1 Sieve Analysis (Mechanical Shaker)

7.3.1.1 Principle

A known mass of extracted, disaggregated sample should be mechanically vibrated through stacked screens with progressively smaller openings. The partial sample weights retained on individual screens are used to produce a sieve analysis report of screen opening size versus percent sample retained.

7.3.1.2 Apparatus

The following equipment is recommended:

a. Mechanical sieving device.
b. U.S. or Tyler sieves (the sieves must conform to ASTM Specification E-11, *Specification for Wire Cloth Sieves for Testing Purposes*).[1]
c. Balance (0.01 gram).
d. Mortar and soft-tipped pestle.
e. Soft bristle brush.
f. Drying oven.

7.3.1.3 Procedure

The screens should be cleaned, dried, weighed, and nested from the largest to the smallest screen size. A pan is placed at the bottom of the screen assembly. Table 7-1 illustrates the commonly available screens and the size equivalents.

A representative core sample is extracted to remove hydrocarbons and salt. The sample is then dried to weight equilibrium to remove solvent and water. The sample is gently disaggregated, weighed to the nearest 0.01 gram (sample size typically 100 grams if eight-inch diameter screens are used),

Table 7-1—Commonly Available Screen Sizes

	U.S. Standard Sieve Mesh	Size in Millimeters
Gravel	5	4.00
Gravel	6	3.36
Gravel	7	2.83
Gravel	8	2.38
Gravel	10	2.00
Very coarse sand	12	1.68
Very coarse sand	14	1.41
Very coarse sand	16	1.19
Very coarse sand	18	1.00
Coarse sand	20	0.84
Coarse sand	25	0.71
Coarse sand	30	0.59
Coarse sand	35	0.50
Medium sand	40	0.42
Medium sand	45	0.35
Medium sand	50	0.30
Medium sand	60	0.25
Fine sand	70	0.21
Fine sand	80	0.18
Fine sand	100	0.15
Fine sand	120	0.13
Very fine sand	140	0.11
Very fine sand	170	0.09
Very fine sand	200	0.07
Very fine sand	230	0.06
Silt	270	0.05
Silt	325	0.04
Silt	430	0.03

and placed into the top screen. The nest of screens is vibrated with a mechanical sieve shaker or manually. The grains travel downward through the nest of screens until retained on a screen having a mesh size smaller than the minimum grain dimensions. Each loaded screen is weighed. The empty screen weights are deducted from the loaded screen weights to obtain the weight of the material retained on individual screens. The data are generally reported as weight percents retained on individual screens and cumulative weight percents to and including each screen. The data are typically reported in both tabular and graphical form (see Table 7-2 and Figure 7-1).

7.3.1.4 Precautions

Precautions of this method include:

a. The sample must be thoroughly extracted to remove residual hydrocarbons. In some cases, salt must be extracted. Care must be taken throughout the extraction process to avoid fines loss.

b. Sample disaggregation must be sufficient to reduce the sample to individual grains without crushing grains or otherwise artificially creating fines.

Table 7-2—Example of Sieve Analysis Data

U.S. Standard Sieve Mesh	Size in Microns	Percent Retained	Cumulative Percent
10	>2000	3.72	3.72
18	>1000	12.69	16.41
35	>500	34.74	51.15
60	>250	21.74	72.89
120	>125	7.30	80.19
170	>88	2.36	82.55
230	>63	3.08	85.63
325	>44	1.55	87.18
430	>30	2.90	90.08
pan	<30	9.92	100.00

c. The screens must be microscopically inspected to ensure that they are not damaged. Puncture-type holes and tears at the screen to retainer contact are particularly troublesome in the finer mesh screens.

d. Sufficient time must be allowed in vibrating the screens to ensure that each size fraction has completely settled. The test duration will vary relative to the quantity and type of sample introduced for a given type of mechanical shaker.

e. The balance must be properly calibrated.

f. In shaly sands, static electricity can prevent charged <.044-millimeter fines from settling to the pan (see 7.3.3 for wet sieve alternates).

7.3.1.5 Calculations

Calculations for this method are as follows:

$$\%\text{Retained Each Screen} = \frac{(\text{loaded screen weight} - \text{empty screen weight})}{\text{total sample weight}} \times 100 \quad (1)$$

$$\%\text{Retained cumulative} = \text{sum of percent retained on each screen (to and including each mesh size from largest screen to pan)} \quad (2)$$

7.3.1.6 Advantages

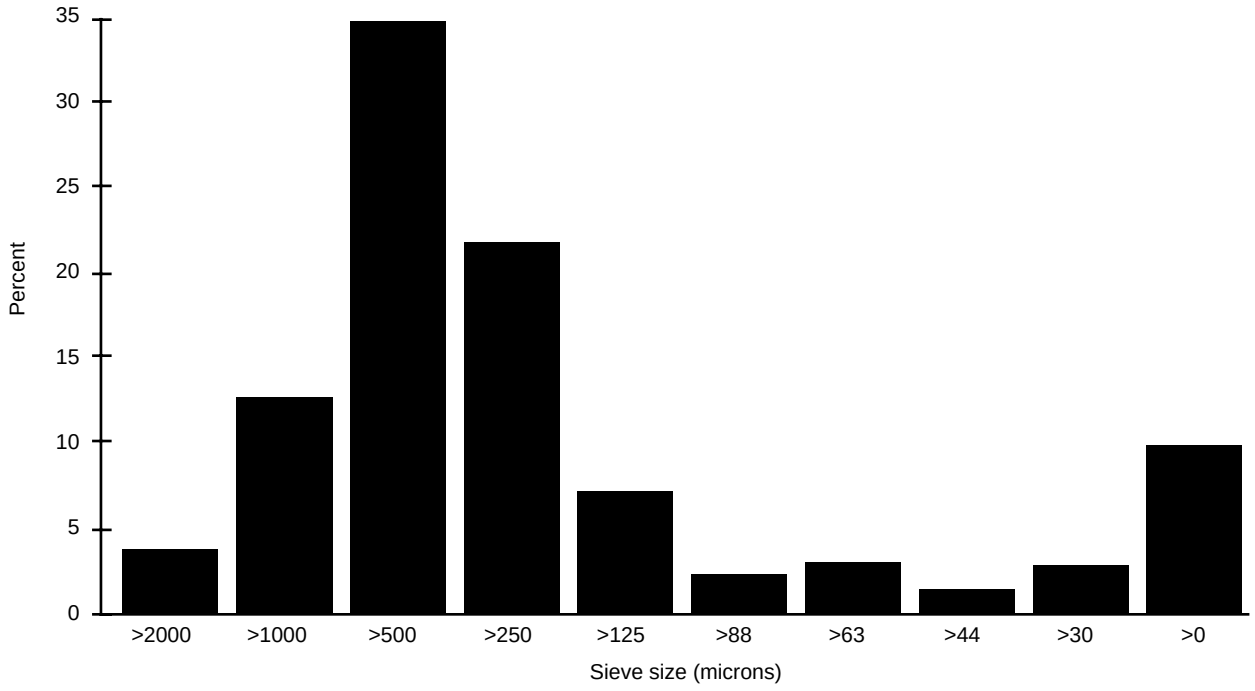
Advantages of this method include:

a. This method is acceptable for classifying grain sizes over the >.044 millimeter size range for minimally cemented sands. The method is a standard method and is accurate.

b. The sample can be recombined for reruns to ensure repeatability.

c. The sample can be recombined or maintained in separate size fractions for additional testing.

d. The test is simple and can be conducted with a minimum of equipment.



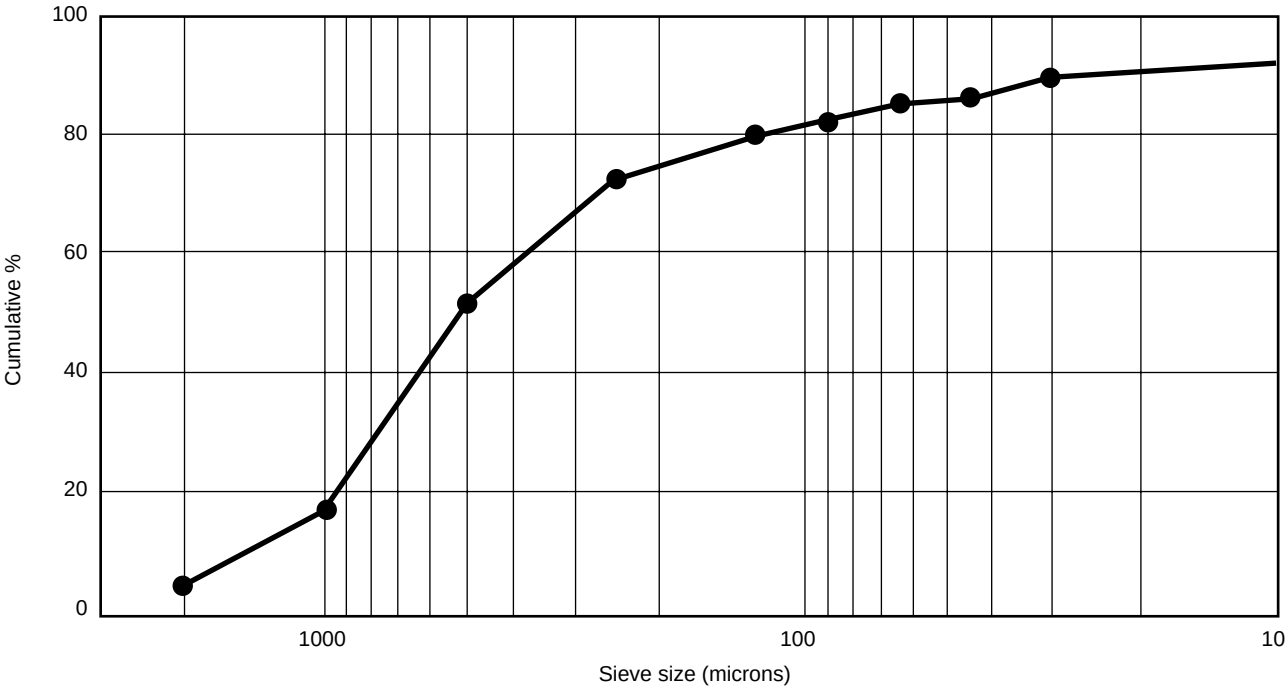


Figure 7-1—Examples of Graphical Forms for Reporting Sieve Analysis Data

7.3.1.7 Limitations

The method is relatively time consuming (one hour per sample for eight-inch diameter screens), requires a relatively large sample, and can yield inaccurate data in shaly sands and consolidated sands.

7.3.1.8 Accuracy and Precision

Provided the test is properly conducted and the precautionary measures above are observed, the expected deviation is less than 3 percent for the total sample and generally less than 0.5 percent for each screen.

7.3.1.9 Calibration

Referenced documents are ASTM C-136, *Standard Method for Sieve Analysis of Fine and Coarse Aggregates*,[2] and ASTM C-702, *Practice for Reducing Field Samples of Aggregate to Testing Size*.[3]

7.3.2 Sieve Analysis (Sonic Shaker)

7.3.2.1 Principle

Refer to 7.3.1, except that properly prepared samples are vibrated through the sieve screens with acoustic energy.

7.3.2.2 Apparatus

The following equipment is recommended:

a. Sonic sieving apparatus.
b. U.S. or Tyler sieves (see 7.3.1.2b).
c. Balance (0.001 gram).
d. Mortar and soft-tipped pestle.
e. Soft bristle brush.
f. Sample splitter.
g. Drying oven.

7.3.2.3 Procedure

Sample preparation and measurement procedures follow the material provided in 7.3.1. Five to fifteen grams of properly prepared sample are placed in the top screen. Sift and pulse amplitudes are adjusted to effectively agitate grains. Sifting time for effective sorting varies from ten to twenty minutes and is dependent upon sample size and degree of shaliness.

7.3.2.4 Precautions

Precautions for this method include:

a. Refer to 7.3.1.4, items a through f.
b. More accurate weight determinations are required than in mechanical sieve analysis because the sample sizes are smaller.

7.3.2.5 Calculations

Refer to 7.3.1.5.

7.3.2.6 Advantages

Advantages of this method include:

a. The method is acceptable for classifying grain sizes over the >0.044 millimeter size range.
b. The test is simple and can be conducted with a minimum of equipment.
c. The test can be conducted more rapidly that the mechanical shaker sieve analysis method (approximately 15 minutes per sample).
d. Less sample is required for the sonic method (5-15 grams) than for the mechanical method.
e. The sample can be recombined for reruns, retained in separate size fractions, or recombined for additional testing.

7.3.2.7 Limitations

Limitations of this method include:

a. Care must be taken to obtain a representative sample due to small sample size.
b. Charged fines retention can be particularly troublesome using this method because the screen frames are plastic. As a result, the sieving time must be increased in shaly sands. In many cases, a significant increase in sieving time may not be adequate to move the <0.044 millimeter fines to the pan.

7.3.2.8 Accuracy

Refer to 7.3.1.8.

7.3.2.9 Calibration

Refer to 7.3.1.9.

7.3.3 Sieve Analysis (Wet and Wet/Dry Combination)

7.3.3.1 Wet sieve analysis refers to the technique of washing a representative, extracted sample through individual sieve screens as opposed to the dry sieving technique, whereby the sample is mechanically agitated or sonified through a nest of sieve screens. The technique requires significantly more time than dry sieving techniques but is preferred in shaly sands.

7.3.3.2 Wet/dry combination sieve analysis, as the name implies, is a combination of both wet and dry techniques. This technique is a very good alternative in shaly sands. The sample is weighed and then wet sieved through the 325 mesh screen to remove the <0.044 millimeter fraction. The material retained on the 325 mesh screen is dried, weighed, and dry sieved in the manner prescribed in the sieve analysis (mechanical or sonic shaker) sections (see 7.3.1 and 7.3.2).

7.3.4 Particle Size Analysis (Laser Diffraction)

7.3.4.1 Principle

An extracted, disaggregated sample is placed into a suitable carrier fluid (carrier fluid may be oil-based or water-based, depending upon test criteria). The carrier fluid is typically stirred or recirculated to ensure particle dispersion. A laser beam is projected through the dispersed sample and the diffracted light patterns are analyzed by microcomputer. The principle upon which grain size distribution is computed is that of Fraunhofer diffraction theory. The angles through which the incident light is scattered are inversely proportional to particle sizes, and the intensity of light scattered by the particles is directly proportional to particle quantities.

7.3.4.2 Apparatus and Supplies

Recommended equipment for this method include:

a. Laser optics device/appropriate computer hardware.
b. Sample splitter.
c. Balance (0.0001 gram), not required for all devices.
d. Mortar and soft-tipped pestle.
e. Sonifier.
f. Deflocculant solution.
g. Drying oven.

7.3.4.3 Procedure

A representative sample is extracted to remove hydrocarbons and dried to remove solvent. The sample is sonified prior to measurement to disperse the particles. The carrier fluid reservoir is charged with carrier fluid and a background measurement is taken. The sample is introduced and surfactant (typically sodium hexametaphosphate) is added to reduce surface tension and to promote particle deflocculation. The carrier fluid/sample mixture is recirculated or stirred sufficiently to ensure uniform particle dispersion. During the measurement phase, a laser beam of known and fixed wave length is projected through the carrier fluid/sample mixture. The forward-diffracted light impacts a photodetector array and Fraunhofer diffraction theory is invoked to compute particle size distribution from the diffracted light flux. The data may be reported in sieve analysis format in tabular and graphical form to the submicron particle size range (see Table 7-2 and Figure 7-1).

7.3.4.4 Precautions

Precautions for this method include:

a. Refer to 7.3.1.4, items a and b.
b. The instrumentation must be properly calibrated (including laser alignment, photodetector array calibration, etc.).
c. The carrier fluid must be bubble-free and particle-free throughout the entire measurement range.
d. The sample must be uniformly dispersed in the carrier fluid and must be deflocculated during the measurement phase.
e. Care must be taken to obtain a representative sample.

7.3.4.5 Calculations

The algorithms to calculate grain size distribution from composite diffraction patterns are complex, manufacturer-dependent, and proprietary. However, the following equations can be utilized to illustrate the relationships between the angular extent of the diffraction pattern, particle size, scattered light intensity, and particle quantity.

The angular extent of the diffraction pattern is given by:

$$\sin\theta = \frac{1.22\lambda}{d} \tag{3}$$

Where:

θ = half-angle to the first minimum of the pattern, degrees.
λ = incident light wave length, microns.
d = particle diameter, microns.

The total intensity of the scattered light is given by:

$$I = Knd^2 \tag{4}$$

Where:

I = total diffracted light, watts.
K = instrument calibration constant, watts/micron^2.
n = number of particles.
d = particle diameter, microns.

7.3.4.6 Advantages

Advantages of this method include:

a. The grain size measurement range provides classification of particles to and including the sub-micron range (very fine silt and clay sized particles).
b. The data are highly reproducible.
c. The technique requires very little sample (typically <1.0 gram).
d. The measurement is rapid (30 seconds to several minutes).

7.3.4.7 Limitations

Limitations of this method include:

a. A size limit exists above which measurements cannot be made. This limit typically falls within the coarse sand fraction. If particle sizes exceed this limit, pre-sieving is required and the data sets must be combined.
b. The instrumentation and associated hardware is expensive.

7.3.4.8 Calibration

Calibration requirements are instrument-manufacturer dependent but generally involve accurate laser beam alignment, photodetector array calibration, and diffraction pattern calibration using a precision standard.

7.3.4.9 Accuracy

Insufficient data exist at present to establish accuracy.

7.3.5 Thin Section Measurement of Grain Size (Visual Technique)

7.3.5.1 Principle

Grain size measurement using thin sections can be undertaken on samples that range from unconsolidated sand to well-consolidated rock. It is the method of choice for consolidated rock, because such rocks cannot be disaggregated successfully without either breaking grains or failing to separate adjacent, well-cemented grains. The technique requires preparation of a thin section and measurement of the size of individual grains visually or with an image analysis system, using a petrographic microscope.

7.3.5.2 Apparatus and Supplies

Recommended equipment for this method includes:

a. Petrographic microscope equipped with an eyepiece micrometer.

b. Thin section preparation equipment.

c. Glass slides.

d. Colored epoxy resin.

7.3.5.3 Procedure

Extracted samples are usually impregnated with colored epoxy resin, mounted on a glass slide, and ground to 30 microns thickness. This thickness allows light to be transmitted through most sand grains. Grain size is measured utilizing the longest axis of individual grain cross sections in the two-dimensional thin section. In many cases, unconsolidated samples need only be dispersed in oil on a glass slide. It is customary to restrict size measurement to a single specific detrital mineral species (usually quartz in sands), although other species may be included (such as feldspar). Typically, 200 to 300 grains are measured. The actual number is dependent upon sorting; poorly-sorted sands require more measurements than well-sorted sands. Grains are selected randomly using the point count technique described by Chayes (1956).[4] Measurements are typically made in millimeters and units are subsequently converted to units of preference (inches, phi units, etc.).

7.3.5.4 Precautions

Precautions for this method include:

a. Thin sections allow for grain size measurement in only two dimensions. Because grains are irregularly shaped and non-uniform in size, and because almost all sediments composed of non-spherical particles show anisotropic dimensional fabrics (i.e., preferred grain orientation), without gross simplifying assumptions there is no unique solution for inferring true grain size distributions determined from thin section measurements (Blatt et al., 1980).[5]

b. Apparatus must be calibrated.

c. Thin sections should be representative of the reservoir interval.

d. Care must be taken to obtain a representative split of the sample because relatively few grains are measured.

e. Sufficient grains must be available for measurement in the thin section to ensure statistical reliability.

f. For laminated sands with contrasting grain size between laminae, unbiased grain size selection can be difficult.

7.3.5.5 Calculations

Grain size is measured using non-dimensional units visible through the microscope eyepiece. These units are scaled to millimeters using a conversion factor dependent upon the magnification used. Size distribution is calculated on a volume frequency basis.

7.3.5.6 Advantages

Advantages of this method include:

a. The measurement range is typically from U.S. Sieve mesh 10 to 400.

b. Thin section measurement of grain size distribution is well suited to most reservoir rocks (sandstones and coarser grained siltstones).

c. The technique allows for standardized measurement of grain size in reservoirs characterized by a range of rock consolidation.

7.3.5.7 Limitations

Limitations of this method include:

a. The technique is tedious, relatively time consuming, and limited to a two-dimensional view.

b. Equipment and instrumentation is expensive.

c. Derivation of mean grain size and the size distribution from the thin section is not straightforward and can be biased. The data are adequate in a relative sense (Kellerhals et al.).[6]

d. Image resolution (magnification and thin section thickness dependent) precludes measurement of very small particles (finer silt and clay size fractions).

7.3.5.8 Accuracy and Precision

The data are slightly biased towards smaller sizes. Precision is a function of the number of grains measured.

7.3.5.9 Calibration

Calibration of size measured in thin section with size measured by sieving and other techniques is possible (Rosenfeld et al. 1953).[7]

7.3.6 Thin Section Measurement of Grain Size (Image Analysis)

7.3.6.1 Principle

Grain size estimates from unconsolidated and consolidated sands can be obtained using automated or semi-automated image analysis procedures. The information provided in 7.3.6 primarily refers to grain size estimates in unconsolidated sands.

7.3.6.2 Apparatus and Supplies

Recommended equipment for this method includes:

a. Image analyzer.

b. Petrographic microscope with light source.

c. Thin section preparation equipment.

d. Glass slides.

e. Colored epoxy resin.

7.3.6.3 Procedure (Unconsolidated Sands)

Sand grains can be dispersed on a glass slide such that few are touching. The projection area or diameter of each grain can be obtained using a simple image analysis apparatus comprised of a videocamera attached to a microscope (Mazullo and Kennedy, 1985).[8] Grains are separated from background using the difference in intensity of the background (brighter) and the grains (darker) as a criterion for thresholding. Typically 200 to 500 grains are required which require 5 to 15 minutes to process.

7.3.6.4 Procedure (Consolidated Sands)

Grain-size distribution using image analysis techniques outlined in 7.3.6 are complicated in consolidated sands by virtue of grain contact. However, techniques exist that utilize the center-to-center distance of intergranular pores to estimate grain size.

7.3.6.5 Precautions

Precautions for this method include:

a. Refer to 7.3.5.4.
b. Inaccurate measurements can occur if the grains to be measured are in contact with one another. This can result in diameters greater than actual.

7.3.6.6 Advantages

Advantages of this method include:

a. Acquisition of raw data is reasonably fast and data are stored in a digital format.
b. Very small samples can be analyzed.

7.3.6.7 Limitations

Limitations of this method include:

a. Care must be taken such that the few hundred grains are representative. This may require careful sample splitting and a careful grain selection procedure on the grain mount.
b. The image acquisition system with software and videocamera is more expensive than the visual technique.

7.3.6.8 Calculations

Data must be converted to volume frequency. This is done by: (a) defining a number of class intervals based on grain diameter, (b) cubing the radii of all grains in each class interval and adding volumes within each interval, and (c) summing all of the class interval volumes to obtain the total volume. The volume fraction in each class interval is calculated by dividing by this total.

7.3.6.9 Accuracy and Precision

The data have been demonstrated to be accurate and deviate only slightly from sieve data (the difference is due to the fact that sieves are shape-selective as well as size-selective).

7.3.6.10 Calibration

Pixel size for a given magnification must be defined when an image analysis system is utilized.

7.3.7 Stoke's Law Particle Size Analysis

7.3.7.1 Principle

A representative, extracted sample is introduced into a liquid column, and the settling velocities of the particles are determined by monitoring the weight increase of a pan suspended in the column. The sedimentation diameter of the particles (the diameter of a sphere of equal density having an equal settling velocity) is calculated using Stoke's Law, a

modified form of Stoke's Law, or Gibbs' formula (Gibbs et al., 1971[9]). The data are typically reported in the same manner as standard sieve data.

7.3.7.2 Apparatus

Recommended equipment for this method includes:

a. Mortar and soft-tipped pestle.
b. Soft bristle brush.
c. Bottom loading balance (0.001 gram) or strain transducer.
d. Settling column and associated computer hardware.
e. Drying oven.

7.3.7.3 Procedure

Sample preparation should follow the protocol outlined in 7.3.1. The sample is precisely weighed (0.001 gram) and introduced into the settling column. The particles fall through the liquid at rates related to size, shape, and density. The particles accumulate on a pan suspended from a precision balance or strain transducer a known distance from the top of the settling column. Weight and time data are acquired by a microcomputer. These data are utilized to compute settling velocities. The test duration varies from several to twenty minutes relative to the type of equipment/instrumentation and the particle size measurement range desired. A modified form of Stoke's Law or Gibbs' formula is typically utilized to calculate sedimentation diameters. If all of the sample is not recovered at the test end point, the weight difference is classified as being finer than the equivalent sedimentation diameter of the last measurement.

7.3.7.4 Precautions

Precautions for this method include:

a. Refer to 7.3.1.4, items a, b, and c.
b. The settling column must be clean (the fluid density must be constant, and suspended particle fallout on a subsequent measurement must be negligible).
c. The particles must be evenly dispersed upon introduction into the column and must be deflocculated.
d. Very careful weighing is required as a result of the small sample quantity used.
e. Convection currents must be eliminated.

7.3.7.5 Calculations

Stoke's Law, a modified form of Stoke's Law or Gibbs' formula are typically used to calculate grain radii from the settling velocity data.

Stoke's Law:

$$V = \frac{2gr^2(\rho_g - \rho_f)}{9\mu} \quad (5)$$

Gibbs' Formula:

$$V = \frac{-3\mu + \sqrt{9\mu^2 + gr^2\rho_f(\rho_g - \rho_f)(0.015476 + 0.19841r)}}{\rho_f(0.011607 + 0.14881r)} \quad (6)$$

Where:

V = settling velocity, cm/sec.
g = acceleration of gravity, cm/sec^2.
r = grain radius, cm.
ρ_g = grain density, g/cc.
ρ_f = fluid density, g/cc.
μ = fluid viscosity, poise.

7.3.7.6 Advantages

Advantages of this method include:

a. The test is rapid (several to twenty minutes).
b. The equipment is relatively inexpensive.
c. The measurement range is relatively broad (through U.S. Sieve 500 mesh).

7.3.7.7 Accuracy and Precision

Information not available.

7.3.7.8 Calibration

The calibration procedure required is described by Gibbs *et al* (1971).[9]

7.4 OIL GRAVITY

The specific gravity of the oil must be known in order to convert oil weight to oil volume in the distillation-extraction method for determining fluid saturations (see 4.3). The oil gravity may be determined by an oil drop method, a refractive index method, a gravimetric method, a digital densitometer method, or a nuclear magnetic resonance method.

7.4.1 Oil Drop Method

7.4.1.1 Principle

The oil drop method consists of suspending a drop of the oil in a liquid medium, the gravity of which can be measured with a hydrometer or specific-gravity balance.

7.4.1.2 Apparatus

Apparatus for this method includes:

a. Glass or clear plastic cylinder.
b. API hydrometer or specific-gravity balance.

7.4.1.3 Procedure

A drop of the oil recovered from the core during the fluid saturation test should be placed in a glass or plastic cylinder

containing a solution of methyl alcohol and water. The gravity of this solution is subsequently adjusted by the addition of alcohol or water until the oil drop remains suspended; i.e., after agitation, the drop neither rises nor falls. The gravity of the solution is then measured with an API hydrometer or a specific-gravity balance. The correct procedure for measuring gravity with a hydrometer is found in ASTM D287-55, *Method of Test for API Gravity of Petroleum.*[10] The observed API gravity at the test temperature is corrected to the gravity at 60°F and converted to specific gravity by the use of Tables 5 and 3, respectively, of the ASTM-IP *Petroleum Measurement Tables* (American Edition).[11] A specific-gravity balance permits a direct reading of gravity. A simple chart for converting units of liquid gravity and density may be useful.

7.4.1.4 Precautions

Precautions for this method include:

a. Any air bubbles adhering to the surface of the oil drop must be removed before adjusting the gravity of the alcohol-water solution.

b. The solution must be agitated after each addition of alcohol or water to ensure a uniform solution and gravity.

7.4.1.5 Advantages

Elaborate equipment is not required.

7.4.1.6 Limitations

Adjustment of the alcohol-water ratio is time consuming.

7.4.1.7 Accuracy and Precision

Information not available.

7.4.2 Refractive Index Method

7.4.2.1 Principle

The refractive index method for determining API gravity is most commonly used to determine the gravity of oil recovered from core samples during the summation of fluids saturation test. The technique is also used to determine the API gravity of stock tank oil, formation test oil, drill stem test oil, etc., in those cases in which very small quantities of oil are available.

A suite of stock tank oil samples of known API gravities for the normally encountered gravity range is required. The refractive indices of the oils, before and after the summation of fluids procedure, are measured. Refractive index versus oil gravity plots are made. Oil gravity can then be determined graphically or calculated using a simple linear regression.

7.4.2.2 Apparatus

Apparatus for this method includes:

a. API hydrometers.
b. Assorted glassware.
c. Glass pipette.
d. Refractometer (refractive index range 1.30-1.90).

7.4.2.3 Procedure

The API gravity of a stock tank oil sample is determined with an API hydrometer using ASTM D287-55: *Method of Test for API Gravity of Petroleum,*[10] or with a specific-gravity balance. A representative aliquot of oil is recovered from the stock tank sample with a glass pipette and the refractometer is charged with the sample. The refractive index of the sample is determined. The stock tank oil sample is then subjected to the retorting phase of the summation-of-fluids analysis (see 4.2). (Retort method for fluid saturation determinations, both sidewall and conventional core analysis.) The refractive index of the recovered oil is determined. Plots of refractive index before and after retorting versus API gravity should be made. API gravities can then be determined graphically or calculated.

7.4.2.4 Precautions

Precautions for this method include:

a. Operation of the refractometer must be verified by measuring the refractive index of a known liquid.
b. The refractometer prism and the glass plate of the refractometer eyepiece must be thoroughly clean.
c. The refractometer prism must be securely placed against the refractometer eyepiece when making measurements.
d. Contaminants such as water and solids must be removed from the oil prior to measurement.
e. The crude must be light-colored and a thin-film refractometer must be utilized.

7.4.2.5 Calculations

A simple linear regression of the stock tank oil API gravities and refractive indices can be made. The refractive index, slope, and intercept can be utilized to calculate the API gravity.

7.4.2.6 Advantages

Advantages of this method include:

a. The technique is simple and rapid, and the data are reasonably accurate.
b. Elaborate equipment is not required.
c. The test requires very little sample (0.03 cc).

7.4.2.7 Accuracy and Precision

API gravity determined using this method should be ±1 degree API of the gravity determined by ASTM D287-55[10], if the calibration is properly made and if the precautionary measures are taken.

7.4.3 Gravimetric Method (Micro-Pycnometer Method)

7.4.3.1 Principle

The density of an oil sample is determined using a small pycnometer and the API gravity is computed.

7.4.3.2 Apparatus

Equipment for this method includes:

a. Balance (0.001 gram).
b. Calibrated pycnometers—The pycnometers can be made of thin-walled glass capillary tubing formed in a U-shape. Various sizes can be prepared by varying the length and/or the inside diameter of the tubing. The volumes should be calibrated with a liquid of known density, with the pycnometer completely full or filled to reference lines (gas-free distilled water may be used).

7.4.3.3 Procedure

A clean, dry pycnometer is weighed. A portion of the oil to be analyzed is drawn into the pycnometer. It is preferable that the largest practical amount of sample be utilized for the gravity determination. The pycnometer is re-weighed. The oil weight is calculated and divided by the capillary volume to determine the oil density. The specific gravity is calculated by dividing the oil density by the water density at the test temperature. The API gravity is calculated.

7.4.3.4 Precautions

The pycnometer weight should be minimized relative to the oil weight. This will reduce error as the weight difference becomes more accurate.

7.4.3.5 Calculations

The following are the calculations for this method:

$$\text{Oil Density (g/cc)} = \frac{\text{charged pycnometer wt (g)} - \text{initial pycnometer wt (g)}}{\text{pycnometer volume (cc)}} \quad (7)$$

$$\text{Specific Gravity} = \frac{\text{oil density (g/cc)}}{\text{water density (g/cc)}} \quad (8)$$

$$\text{API Gravity (deg)} = \frac{141.5}{\text{specific gravity}} - 131.5 \quad (9)$$

7.4.3.6 Advantages

Advantages of this method include:

a. Accurate data can be obtained if the pycnometer has been properly calibrated.
b. The determination is rapid.

7.4.4 Digital Densitometer Method

7.4.4.1 Principle

The measurement principle of the digital densitometer is based on the change of the natural frequency of a hollow oscillator when fluid-filled. The oscillator frequency change is related to the mass change of the oscillator subsequent to sample introduction, and as a result, to the sample density.

7.4.4.2 Apparatus

A digital densitometer is required.

7.4.4.3 Procedure

Instrument calibration constants are determined by measurement of the air-filled and distilled water-filled oscillator frequencies. The sample is injected into the oscillator and the oscillator frequency is determined. The API gravity is calculated as indicated in 7.4.3.5.

7.4.4.4 Advantages

The technique is rapid and accurate.

7.4.4.5 Limitations

Relatively large amounts of oil are required (1 to 2 cc).

7.4.5 Nuclear Magnetic Resonance (NMR) Method

7.4.5.1 Principle

The NMR method for determining the viscosity of crude oils is based on Stoke's relationship between viscosity and molecular translational and rotational diffusion that cause NMR spin-lattice relaxation. Either ^{1}H or ^{13}C NMR can be used. A major advantage is that the method can be applied to bulk crude oil and/or oil/water emulsions, crude oil inside core plugs or whole cores, or crude oil dissolved in solvent extracts.

7.4.5.2 Apparatus

Fourier Transform NMR Spectrometer with ^{1}H chemical shift or ^{13}C capabilities. For NMR diffusion measurements, a pulsed gradient NMR probe is required with field gradients above 100 gauss/cm.

7.4.5.3 Procedure

The ^{1}H or ^{13}C spin lattice relaxation time (T1) is measured with an inversion recovery or saturation recovery pulse sequence. Viscosity and API gravity are then determined from published correlations. ^{13}C NMR is preferred for analysis of oils inside cores because chemical shift separation between oil and water phases in the core is not required.

The application of a large, steady-pulsed magnetic field gradient enables direct ^{1}H NMR measurements of translational diffusion in liquids. The pulse sequence is normally a 90°-T-180°-spin echo with magnetic field gradient pulses applied after the 90° and 180° RF pulses. Diffusion in an applied field gradient causes an irreversible loss in the spin echo amplitude because only the nuclei that do not diffuse to a region with a different magnetic field will be refocused. The interpulse time (T) is kept very short (approximately 1 millisecond) to prevent restricted diffusion at the pore walls, and the water and oil signals are separated either by chemical shift spectroscopy or by doping the water with Mn-EDTA to eliminate the water signal. The oil diffusion coefficient can be related to viscosity via Stoke's Law and API gravity can be estimated from published correlations between viscosity and API gravity.

7.4.5.4 Advantages

Advantages of the NMR method include:

a. The technique is rapid, noninvasive, and can be automated.
b. The technique is applicable to bulk oil, oil in plug cores, or whole core, and liquid extracts.
c. Minimal sample preparation is required.
d. Measurements can be made on oil/water emulsions.
e. Very small amounts of oil are required for ^{1}H NMR (<0.1 cc).

7.4.5.5 Limitations

Limitations of the NMR method include:

a. The NMR spectrometer is expensive.
b. The T1 method is applicable for oils only up to approximately 1,000 cp (approximately 15 degrees API) because of the flattening of the T1 versus viscosity correlation. This limitation does not apply to NMR diffusion measurements, which are only limited by the strength of the applied field gradient.

7.5 OIL CHARACTERIZATION

7.5.1 Oil Characterization (Chromatographic Method/Hydrocarbon Vaporization)

Residual hydrocarbons entrained in drill cuttings and core samples can be analyzed to quantify hydrocarbon volume and to evaluate the compositional characteristics of the oil. These data can be interpreted to provide information in the following areas:

a. Alteration processes—The crude oil quality is dictated by one or more of the alteration processes that affect the oil during, or subsequent to, its accumulation in a reservoir. The changes resulting from alteration processes are reflected in the composition of the residual hydrocarbon found in the rock pore spaces.
b. Type of hydrocarbon production—The composition of the residual hydrocarbon in the rock matrix at the surface reflects the original reservoir composite less components that have escaped through pressure depletion. The distribution of intermediate and heavier components can be used to predict the type of hydrocarbon production (non-productive, gas, condensate, or oil).
c. Changes in rock characteristics (permeability)—The escape of components during pressure depletion is limited by molecular size and hydrocarbon volatility, but is also greatly influenced by permeability. Compositional contrast can often be related to permeability and permeability variation.
d. Contamination (drilling fluids and/or additives)—Drilling fluids and/or additives can be analyzed by the same procedure as the rock samples. Compositional contrast and hydrocarbon ratio techniques can be utilized to distinguish indigenous hydrocarbons from drilling contaminants.
e. Correlation of produced oil to drill cuttings or core sample oil.

7.5.1.1 Principle

This method involves vaporization of residual hydrocarbons in drill cuttings or core samples directly onto a chromatographic column. The components separated by the column are detected by a flame ionization detector (FID). Peak heights and areas are obtained from the recorded output.

7.5.1.2 Apparatus

Apparatus recommended for this method includes:

a. Chromatograph—The chromatograph should contain a large bore capillary column (0.5 millimeter ID) containing a stationary phase that is suitable for separation and a flame ionization detector.
b. Vaporization cell (low dead volume, accurate temperature control, and even temperature distribution).
c. Sample boat (quartz tube).
d. Integrator.
e. Balance (0.001 gram).

7.5.1.3 Procedure

A representative sample (0.01 to 1.0 gram) should be selected and placed in a tared quartz sample boat (drill cuttings must be rinsed with water to remove drilling fluid and

air dried for two to four hours prior to charging the boat). The quantity of sample used is dependent on the oil saturation, the detector capacity, and the column limitations. The charged sample boat is weighed and placed in the vaporization cell. Human skin oil is a possible contaminant and care should be taken to avoid directly handling the sample.

The hydrocarbons are vaporized at a temperature that will not induce cracking. Temperatures above 320°C will cause cracking of larger molecules. The gas evolved is passed directly onto the column without splitting. Resolution can be enhanced by increasing the column temperature at a programmed rate. Output of the flame ionization detector can be converted to millivolts (height) and millivolts x seconds (area). Specific peaks are identified by their retention time.

7.5.1.4 Precautions

Precautions for this method include:

a. The sample must be representative.

b. Large samples may increase reproducibility but will restrict peak resolution.

c. High bound-water saturation may cause a suppression of detector response, particularly in the lighter midrange components.

d. Accurate weights are required (±0.001 gram).

e. Care must be taken to avoid heating during sample drying to prevent lighter component losses.

7.5.1.5 Calculations

7.5.1.5.1 Quantification of residual hydrocarbon volume:

$$\text{Normalized total area } (A_{nt}) = \frac{\text{total area } (A_t)}{\text{sample weight } (g)} \quad (10)$$

7.5.1.5.2 Alteration processes—Indices can be tabulated to distinguish changes in composition.

Following are several indices that are typically used to determine type, origin, and alteration processes:

a. n-C15+ (%) = $\dfrac{> n\text{-C15 area}}{A_t} \times 100$ (11)

b. n-alkane (%) = $\dfrac{n\text{-c components area}}{A_t} \times 100$ (12)

c. ratio n-C17 + n-C18 to pristane+phytane =

$$\frac{(\text{height n-C17} + \text{height n-C18})}{(\text{height pristane} + \text{height phytane})} \quad (13)$$

d. pristane/phytane ratio = $\dfrac{\text{height pristane}}{\text{height phytane}}$ (14)

e. Carbon preference index =

$$\left|\frac{\Sigma\text{odd heights n-C17-C31}}{\Sigma\text{even heights n-C16-C30}}\right| + \left|\frac{\Sigma\text{odd heights n-C17-C31}}{\Sigma\text{even heights n-C18-C32}}\right| \quad (15)$$

7.5.1.5.3 Type of hydrocarbon production:

The area percent within each carbon group (light, intermediate and heavy) is calculated as follows:

a. % light components = $\dfrac{< \text{C8 area}}{A_t} \times 100$ (16)

b. % intermediate components = $\dfrac{< \text{C13 area}}{A_t} \times 100$ (17)

c. % heavy components = $\dfrac{> \text{C18 area}}{A_t} \times 100$ (18)

Prediction of hydrocarbon production can be based on the following general guidelines:

a. Non-hydrocarbon productive—relatively high percentage of lighter components, relatively low total area.

b. Gas productive—relatively high percentage of lighter components with lower percentages of intermediate components, relatively high total area.

c. Oil productive—relatively high percentage of intermediate and heavy components, high total area.

7.5.1.6 Advantages

Advantages of this method include:

a. Extremely small samples (0.01-1.0 gram) can be utilized.

b. Solvent extraction and sample splitting are not required.

c. Oil composition and residual hydrocarbon content can be monitored simultaneously.

7.5.1.7 Limitations

Bound water may suppress detector response, particularly in the lighter midrange components.

7.5.1.8 Accuracy

Accurate sample weights and careful control of operating conditions should yield oil volumes reproducible to within 3 percent. Reproducibility of component distribution is dependent on maintenance of operating conditions and column condition and can be verified by repeat runs.

7.5.1.9 Calibration

Equipment calibration is essential for consistent results. The following areas are critical:

a. Component identification—Identification of individual peaks is based in part on retention time. Retention time is dependent on carrier gas flow rate, column temperature, and the condition of the column stationary phase. Carrier gas flow rate should be calibrated and the system should be leak tested routinely. Retention times for specific peaks can be measured. A sample can be spiked with specific components, such as the normal alkanes for calibration purposes.
b. Calibration of detector response—A standard should be used to check peak resolution and system noise. Relative areas should be consistent. Relative standard deviation of 2 to 3 percent is typical.
c. Reproducibility—Reproducibility can be evaluated by making repeated runs of the same sample. Specific indices used should be checked along with total area normalized by weight.

7.6 ACID SOLUBILITY

7.6.1 Principle

Acid solubility tests on drill cuttings or core samples are typically used to assess HCl and/or mud acid (HCl/HF mixture) reactivity with reservoir rock. These data are used to evaluate the potential success of acid stimulation, near wellbore damage removal, to assess formation damage sensitivity, to determine calcite/dolomite ratios in carbonates, and to determine quartz content in carbonates. Test procedures can be adapted to determine acid volumes required for field treatment and to determine acid response coefficients.

The acid solubility test is a gravimetric test performed to determine formation sensitivity to acid. A known mass of extracted and dried sample is placed in excess acid for a specific residence period. Subsequently, the sample is dried and reweighed. The weight difference is used to calculate the percent acid-soluble material.

7.6.2 Apparatus and Supplies

Equipment for acid solubility determination includes:

a. Balance (0.001 gram).
b. Assorted glassware for HCl solubility (beakers, funnels, graduated cylinders, etc.).
c. Assorted polyethylene or polypropylene beakers, funnels, graduated cylinders, etc. for mud acid solubility.
d. Mortar and pestle.
e. U.S. Sieve, 80 mesh screen.
f. Drying oven.
g. Acid-resistant filter paper.
h. HCl, 15 weight percent.
i. Mud acid (HCl, 12 weight percent—HF, 3 weight percent).
j. Appropriate safety equipment (face shield, apron, etc.).

7.6.3 Procedure (HCl Solubility, Calcite Content)

A representative sample is extracted to remove hydrocarbons and dried to constant weight to ensure removal of solvent and water. The sample is disaggregated sufficiently to produce several grams of material finer than U.S. Sieve 80 mesh. Approximately 1 gram of sample is weighed and placed in 150 cc of 15 percent HCl for a minimum of 60 minutes and a maximum of 65 minutes without stirring. The filtration apparatus is prepared and the filter paper is weighed. The acid/sample mixture is filtered and rinsed with deionized water. The remaining sample is dried and weighed. The weight difference is used to compute the percent acid soluble material.

7.6.4 Procedure (HCl Solubility, Calcite Plus Dolomite Content)

Dolomite is also soluble in HCl at 80°C. Applying the procedure in 7.6.3 at this temperature yields a value referred to as the total carbonate content (calcite plus dolomite).

7.6.5 Procedure (Mud Acid Solubility, Total Acid-soluble Content)

Carbonates and siliceous materials such as feldspars and clays dissolve in a mixture of HCl and HF. This mixture is generally called mud acid. Although various concentrations are in use, a mixture of 12 weight percent HCl and 3 weight percent HF is most commonly applied. Applying the procedure above, using HF tolerant apparatus, with mud acid results in the total acid solubility (i.e., carbonates, feldspars, iron oxides, clays, etc.).

7.6.6 Precautions

The procedure is gravimetric and requires strict adherence to procedures and good laboratory technique to provide reproducibility.

CAUTION: In all procedures outlined in 7.6.3, 7.6.4, and 7.6.5, appropriate safety precautions for handling acids must be employed.

7.6.7 Calculations

The following are the calculations for this method:

Calcite Content (HCl, room temperature):

$$\% \text{ Acid soluble} = \frac{\text{initial sample weight} - \text{final sample weight}}{\text{initial sample weight}} \times 100 \quad (19)$$

Total Carbonate Content (HCl, 80°C.):

$$\% \text{ Acid soluble} = \frac{\text{initial sample weight} - \text{final sample weight}}{\text{initial sample weight}} \times 100 \quad (20)$$

$$\% \text{ Dolomite} = \% \text{ total carbonate content} - \% \text{ calcite content} \quad (21)$$

Total Acid Soluble Content (Mud acid):

$$\% \text{ Acid soluble} = \frac{\text{initial sample weight} - \text{final sample weight}}{\text{initial sample weight}} \times 100 \quad (22)$$

7.6.8 Advantages

The technique is uncomplicated and requires simple equipment.

7.6.9 Limitations

Limitations of this method include:

a. The procedure is indicative of maximum rock reactivity, as the sample is disaggregated and the test is conducted in excess acid.

b. Siderite is soluble in HCl at room temperature and, as a result, can distort the calculated calcite content.

7.6.10 Accuracy and Precision

Information not available.

7.6.11 Calibration

The balance must be properly calibrated.

7.7 CORE WATER SALINITY DETERMINATION

A salinity determination on the water present in the core can be desirable because it could aid in core analysis data interpretation and in electric log evaluation.[12,13] Salinity or total dissolved solids (TDS) is defined as the sum of all dissolved materials in terms of milligrams of salt per kilogram of solution (mg/kg) which is equivalent to parts per million (ppm). Salinity is often calculated from the amount of chloride ion present in the core water and is expressed as the equivalent amount of sodium chloride. However, if the salinity is calculated from a resistivity measurement, it represents an NaCl equivalent of the contribution from all soluble electrolyte ions. Measurements of the core water salinity are based on the assumption that all of the chemical ions and gases in the core formation water at reservoir conditions are still contained in the core water at ambient conditions. This may not be the case for brines with ion concentrations near the solubility limits, i.e., soluble at reservoir temperature and pressure but insoluble at ambient conditions.

During the coring operation, the coring fluid (mud) filtrate may invade the core and dilute the formation water. There are three factors to minimize mud filtrate diluting the formation water. The core material should be obtained using a low invasion coring bit, the coring fluid should have a low API fluid loss, core samples for analysis should be taken at well site right after the core surfaces.[14,15,16] The core plugs for core water salinity should be taken from the center of the core, farthest from the perimeter of the core where filtrate invasion will be the greatest (see 4.3.7).[15] To avoid evaporation of water from the core/plug material, the core/plug should be sealed immediately after retrieval (see 2.5). Freezing the whole core after removal from the core barrel to prevent tracer migration and counter-current imbibition is not a recommended alternative to taking a plug sample at well site from the middle of the core material. A salinity front develops as the core freezes which concentrates salts ahead of the freeze line.[17]

If the salinity of the formation water and the mud filtrate are known, the degree of flushing by the coring fluid can be estimated by the core water salinity (but may be affected by nonuniform distributions). Even if the salinity of formation water is not known, the degree of flushing by the water based coring fluid may be estimated if the coring fluid contains a tracer chemical; such as tritiated water, bromide, or iodide, that can be assumed to be at a low concentration in the formation water (see 4.3.7).[18]

If the coring fluid contains a high concentration of potassium, because potassium chloride is the main source of salinity in the coring fluid, then the potassium ion can be used as the tracer. In formation waters, the ratio of sodium to potassium is much greater than one. A cation analysis (sodium and potassium) of the core water provides a method to determine if the core is relatively free of coring fluid. The use of potassium ion as a tracer can only provide a qualitative indication of the extent of coring fluid filtrate invasion compared with the more quantitative assessment using coring fluid tracer chemicals; such as tritiated water, bromide, or iodide. These anions are not chemically attenuated by the rock matrix as is possible for cations like potassium.

7.7.1 Core Water Recovery

To measure salinity of the core water, it must first be separated from the core. This can be achieved by mechanically expelling the core water from the core using an immiscible fluid flush or by centrifugation. When the core water is near irreducible saturation, mechanical expulsion may be unsuccessful. When no brine is expelled by mechanical means, then the salts can be water-extracted from a dried core. The salinity can then be estimated from the amount of salt extracted and the amount of water initially present in the core.

7.7.1.1 Centrifugation

7.7.1.1.1 Principle

Core fluids, including the formation water, may be expelled by centrifuging the sample.

7.7.1.1.2 Apparatus

Recommended apparatus for this method includes:

a. Centrifuge and sample holders.
b. Assorted glassware.

7.7.1.1.3 Procedure

The centrifuge should be of such a size and so driven that a force equal to at least 1,000 times the force of gravity can be exerted on the center of gravity of the core sample. The centrifuge sample holder should be an airtight unit having a perforated bottom with a receiving chamber. Unconfined core samples are placed in weight-matched centrifuge sample holders. The samples are centrifuged for at least one hour, preferably overnight if the sample permeability is less than 100 md. A sample of the produced brine is separated from the fluid mixture centrifuged from the core, with the use of a separator funnel or by pipetting a sample from the brine layer. This sample should be saved for salinity determination.

7.7.1.1.4 Precautions

Precautions for this method include:

a. The temperature of the centrifuge should be controlled to reduce the potential for water loss from the sample holders.
b. Care should be taken to avoid evaporation of water during all steps, starting with handling the core material to transferring of the brine sample to the sample container for salinity determination.

7.7.1.1.5 Advantages

Advantages of this method include:

a. The core sample does not have to have a cylindrical shape or be consolidated.
b. The presence of reservoir crude oil in the core sample does not interfere with this procedure or the validity of the results.

7.7.1.1.6 Limitations

Limitations of this method include:

a. The time required to centrifuge out the brine may be long for low permeability samples.
b. The procedure only works if the core contains a high enough brine saturation such that a portion can be removed at the highest available centrifuge speed.
c. The centrifugal force on the unconfined sample may irreversibly alter the matrix of the rock, changing porosity and permeability, such that the core may not be suitable for subsequent core analysis tests.

d. The salinity of the core water expelled may not be representative of the core water salinity at reservoir pressure and temperature conditions.

7.7.1.1.7 Accuracy/Precision

The accuracy/precision of the separation technique is limited by the ability to preserve core from well site to the laboratory and the subsequent handling of the core samples and the extracted brine sample without altering the salinity of the core water. Evaporation effects for relatively small samples or samples containing very low brine saturations, Sw=<5%, may increase salinity. Percent error will be significantly reduced as the amount of core water volume increases. Careful core handling practices may result in errors in core salinities due to core and brine handling being less than 2% of total salinity.[13]

7.7.1.2 Immiscible Fluid Flush

7.7.1.2.1 Principle

The formation water may be expelled by an immiscible displacement flush.

7.7.1.2.2 Apparatus

Apparatus for this method includes:

a. Hassler or triaxial core holder.
b. Solvent pump.
c. Assorted glassware.

7.7.1.2.3 Procedure

A core plug is mounted in a rubber sleeve Hassler or triaxial core holder cell that will permit fluid to flow through the matrix of the sample. Details of this procedure are given in 3.6.4.1. The water-immiscible fluid could be a refined mineral oil or an organic solvent like toluene. The higher the viscosity contrast between the water-immiscible fluid and water, the more likely that some formation water will be produced during the flush. A sample of the produced brine is separated from the fluid mixture flushed from the core using a separator funnel or by pipetting a sample from the brine layer. This sample should be saved for salinity determination.

7.7.1.2.4 Precautions

Precautions for this method include:

a. The fluid used to expel the formation water should have a low solubility for water so as not to change the salinity of the extracted water.
b. Care should be taken to avoid evaporation of water during handling of the core, flushing, and transferring of the brine to the sample container for salinity determination.

7.7.1.2.5 Advantages

The presence of reservoir crude oil in the core sample does not interfere with this procedure or affect the validity of the results.

7.7.1.2.6 Limitations

Limitations of this method include:

a. For low permeability samples, the time required to flush out the brine may be long.
b. This procedure only succeeds if the core contains a brine saturation which can be reduced by the available applied pressure drop, e.g., core is from water or oil-water transition zone.
c. If the net hydrostatic pressure used to confine the sample while flushing exceeds the net reservoir overburden pressure, the sample porosity may be irreversibly compacted and the sample may not be suitable for subsequent core analysis tests.
d. The core sample must have cylindrical shape.
e. The salinity of the core water expelled may not be representative of the core water salinity at reservoir pressure and temperature conditions.

7.7.1.2.7 Accuracy/Precision

See section 7.7.1.1.7.

7.7.2 Water Extraction of Core Salt

Core water salts are extracted from a dried core sample.

7.7.2.1 Apparatus

Apparatus for this method includes:

a. Mortar and pestle.
b. Balance.
c. Desiccator.
d. Assorted glassware.

7.7.2.2 Procedure

Approximately 50 grams of sample are selected. The water saturation must be known. The preferred method is to use a portion of the sample used in a previous saturation determination test. Less preferably, the sample may be selected from core material at a point as close as possible to the sample used in the saturation measurement test.

The sample must be free of hydrocarbons. Core material from a Dean-Stark toluene extraction is suitable. The sample is ground in a mortar to approximately 16-mesh size and dried in an oven to constant weight. After being cooled in a desiccator, the sample is weighed and transferred to a flask. 100 ml of distilled water is added and the mixture is stirred vigorously for several minutes. Agitation is continued intermittently for a minimum of one hour. The resulting salt solution is filtered or decanted and saved for salinity determination.

7.7.2.3 Precautions

Precautions for this method include:

a. If the core sample is suspected of containing clay material, a constant humidity oven should be used at a temperature of about 140°F (60°C) and 45 percent humidity during the drying step for determining core water content. This is done to avoid removing chemically- and physically-bound water (water that does not contribute to the salinity),[19] and thus to avoid overstating the amount of core water, which results in underestimating the core water salinity. Since constant humidity drying under these conditions does not dehydrate clays, no detectable fraction of the 100 ml of water added to the sample will be used to rehydrate the clays.
b. Care should be taken to avoid evaporation of water during all steps, starting with core preservation to transferring of the brine sample to sample container for salinity determination.

7.7.2.4 Advantages

This procedure is simple and can be conducted with very little equipment in a short time.

7.7.2.5 Limitations

Limitations of this method include:

a. The presence of hydrocarbons in the core sample interferes with the determination of core water content. The more volatile components of the hydrocarbons will be lost during the drying step, and thus the amount of water present will be overstated.
b. If the core material contains any sulfide minerals, such as pyrite that has oxidized during core handling, storage, or during extraction, the amount of salts extracted will be overstated. Oxidation of pyrite will produce water soluble iron sulfate salts.
c. If the in situ core rock matrix includes solid phase soluble salts (that are in solubility equilibrium with the formation brine), i.e., anhydrite or sodium chloride, then during drying and extraction these salts may be extracted because of the

high water to rock ratio during extraction, and the salinity of the core water will be overstated.

d. The sample is destroyed.

e. The calculated salinity of the core water may not be representative of the core water salinity at reservoir pressure and temperature conditions.

7.7.3 Determination of Core Water Salinity

The salinity of the water is determined, using either: (a) chemical determination of the chloride ion, (b) refractive index, or (c) resistivity measurement. The salinity is expressed as parts of sodium chloride per million parts of core water (although other salts are usually present), which is equivalent to milligrams of sodium chloride per kilogram of solution (sodium chloride and water).

There are several other analytical techniques for measuring the salt content of the core water. These include ion chromatography, atomic absorption, and inductively coupled plasma spectroscopy. A combination of these methods can accurately determine the cationic and anionic composition of core water, but the equipment is much more expensive than that required by procedures described in 7.7.3.1, 7.7.3.2, and 7.7.3.3.

7.7.3.1 Chemical Determination of Chloride Ion

The chloride concentration is measured in the undiluted brine solution expelled from the core or the filtrate obtained by separation of the crushed sample and the water used in the leaching process. The salinity of the water is expressed as parts per million sodium chloride in the core water (mg/kg).

7.7.3.1.1 Apparatus and Procedure

The apparatus, reagents, and procedure for chloride determination are described in detail in API RP 45,[20] *Recommended Practices for Analysis of Oilfield Waters*, and also in ASTM D512-89,[21] *Standard Test Methods for Chloride Ion in Water*. Of the four ASTM methods listed, Method A (Mercurimetric Titration), Method B (Silver Nitrate Titration), Method C (Colorimetric), and Method D (Ion-Selective Electrode), Method B is preferred. The calculation described in this reference will give the salinity in terms of chloride ion instead of sodium chloride. The calculation as sodium chloride is described in 7.7.3.1.3.

7.7.3.1.2 Precautions

Precautions are as follows:

a. Brine sample should be diluted to fall within optimum range of the titration solutions and equipment.

b. Make blank chloride determinations to correct for any chloride present in the glassware and water used in the tests.

7.7.3.1.3 Calculations

The following calculations are used:

a. The salinity of the core water expelled from the core sample can be calculated directly. The normality of salt solution measured by a chloride titration can be converted to sodium chloride concentration, mg/kg, as follows:

$$\text{Conc. of NaCl (mg/kg)} = 1{,}000 \times N_1 \times MW_{NaCl}/D_w \qquad (23)$$

Where:

$$N_1 = V_2 \times N_2/V_1 \qquad (24)$$

N_1 = normality of salt solution titrated, meq/ml.
V_1 = volume of salt solution titrated, ml.
N_2 = normality of chloride reagent (i.e., silver nitrate), meq/ml.
V_2 = volume of chloride reagent at titration endpoint, ml.
D_w = density of core water, gm/ml.
MW_{NaCl} = equivalent weight of NaCl = 58.5 gm/equ.

b. When core water cannot be expelled and the salts are water extracted, the salinity of the originally-present core water must be back-calculated. The salinity from the chemical determination method is converted to total milligrams of sodium chloride leached from the sample. This number represents the total salt from the core water of the sample. The total salt in milligrams and the weight of the volume of core water obtained by saturation tests are used to calculate the concentration of salt in the core water, and this value is expressed in parts per million of sodium chloride.

The milligrams of sodium chloride leached from the core sample determined from the chloride titration are calculated as follows:

$$\text{Weight of NaCl (mg)} = N_1 \times 58.5 \times V_w \qquad (25)$$

Where:

$$N_1 = V_2 \times N_2/V_1 \qquad (26)$$

N_1 = normality of salt solution titrated.
V_1 = volume of salt solution titrated, ml.
N_2 = normality of chloride reagent (i.e., silver nitrate).
V_2 = volume of chloride reagent at titration endpoint, ml.
V_w = volume of water used to leach or extract the sample, ml.

The weight of the core water can be obtained directly by difference between the weight of the sample before and after drying, if no oil is present. The drying should follow the directions as given in 5.3.2.2.3.6.

The salinity (mg/kg) of the core water is obtained by:

$$\text{mg NaCl/kg of core water} = \frac{\text{mg NaCl}}{\text{(gm core water + gm NaCl)}} \times 1000.0 \qquad (27)$$

If the sample contains oil, the following calculations can provide an approximate salinity value. The fluid saturations and the porosity must be determined either on this sample or an adjacent core. The amount of water contained in the sample can be calculated as follows:

$$\text{Weight of core water, gm} = V_b \times \emptyset \times S_w \times D_w \quad (28)$$

Where:

$$V_b = [(W/D_g)/1 - \emptyset)] \quad (29)$$

W = dry weight of sample, gm.
D_g = grain density, gm/cc.
$\emptyset$ = porosity, as fraction.
V_b = bulk volume of sample, cc.
S_w = water saturation, as fraction of pore volume.
D_w = density of core water, gm/ml.

If the grain density has not been determined, use a representative value for the formation of interest. The density of the core water is assumed to be 1.00 as an initial estimate. After the salinity is calculated with Equation 27, the weight of the core water, Equation 28, is recalculated using a new density based on the previously-calculated salinity; and a new salinity is recalculated using Equation 27.

7.7.3.1.4 Advantages

Advantages of this method are as follows:

a. Method is rapid and convenient.
b. Chloride determination is accurate.

7.7.3.1.5 Limitations

Limitations of this method include:

a. Result is expressed as sodium chloride. Anions other than chloride, such as sulfate and bicarbonate, present in the solution are not detected.
b. Other halides, such as bromide and iodide, are determined as chloride.

7.7.3.1.6 Accuracy/Precision

When the core water can be recovered from the core by centrifugation or an immiscible flush, the accuracy in the determination of salinity (mg/kg) is controlled by the salinity determination technique, chloride titration, on the core water sample which is about ±1 to 2% of the total salinity. API Recommended Practice 45 reports a precision of about 1% and an accuracy of about 2% of the amount present. When the core water salts have to be extracted from a dried core, the accuracy in the determination of core water salinity (mg/kg) is limited by the determination of the water content.[13] McCoy, et al.[13] report field data where chloride salinity determinations on oil-based-mud (OBM) cores intersecting the oil-water contact agreed within 1% on the average with produced water salinity. Chloride concentration measurements were obtained on the water extracts of about 2,000 OBM dried core samples (using procedures similar to 7.7.2 and 7.7.3).

7.7.3.2 Refractive Index Measurement

The total ionic constituents of water may be estimated by measuring the refractive index. The refractive index varies in a direct manner with the ionic concentration of sodium chloride and other salts. Standard graphs, which show the refractive index value for various salinities and temperatures of sodium chloride solutions, can be prepared from data in the literature (see Figure 7-2).[22,23] If the refractive index is known, the salinity of the core water, expressed as parts per million of sodium chloride (mg/kg), can be determined from such a graph using a measured refractive index value.

7.7.3.2.1 Apparatus

Apparatus for this method includes:

a. Refractometer.
b. Assorted glassware.

7.7.3.2.2 Procedure

The instrumentation should be calibrated with known concentrations of sodium chloride solutions, resulting in a graph similar to that shown in Figure 7-2. Details on apparatus and procedures may be found in ASTM D542-85, *Standard Test Method for Pore Water Extraction and Determination of the Soluble Salt Content of Soils by Refractomer.* The refractive index is measured on brine sample. With the use of a standard graph (see Figure 7-2) and suitable calculations, the refractive index value is converted to a salinity value for the core water.

7.7.3.2.3 Precautions

Refractive index value must be corrected to a standard temperature.

7.7.3.2.4 Advantages

Advantages of this method include:

a. Rapid determination.
b. The total equivalent sodium chloride concentration is determined.

7.7.3.2.5 Limitations

All ions present in the water are calculated as sodium chloride.

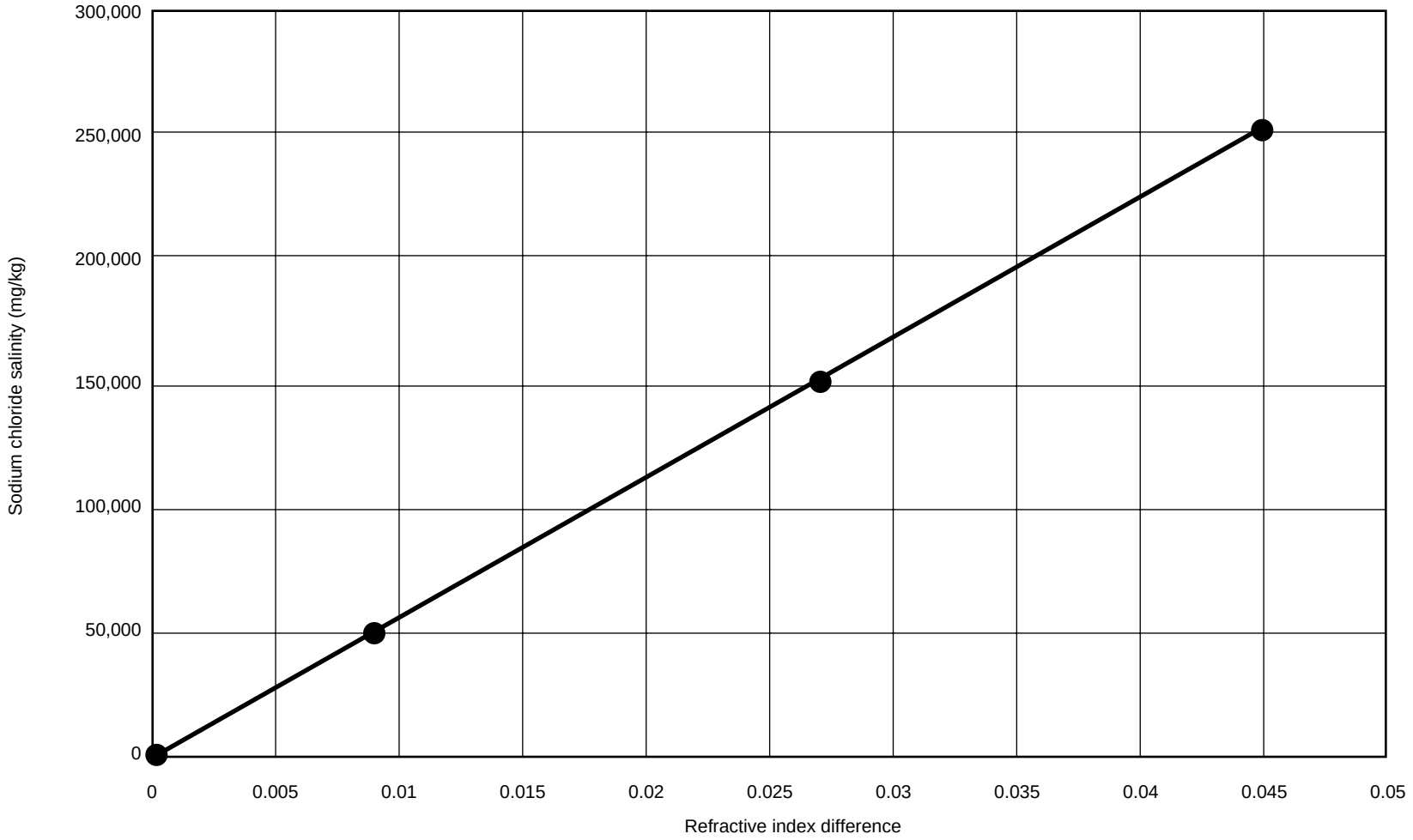


Figure 7-2—Refractive Index (RI) Difference Versus Salinity (RI of Solution—RI of Distilled Water)

7.7.3.2.6 Accuracy/Precision

See 7.7.3.1.6 for general comments. According to ASTM D4542, salinity determination using the refractometer should be accurate within ±300 mg/kg salinity.

7.7.3.3 Resistivity Measurement

The total ionic constituents of water may be estimated by measuring the resistivity. The resistivity varies in an inverse manner with the ionic concentration of sodium chloride and other salts over a wide range of concentrations. Standard graphs, illustrated by Figure 7-3, show the resistivity value for various salinities and temperatures of sodium chloride solutions. If the resistivity is known, then the salinity of the core water, expressed as parts per million of sodium chloride (mg/kg), can be determined from such a graph using a measured resistivity value.

7.7.3.3.1 Apparatus

Apparatus for this method includes:

a. Resistivity cell.
b. Resistivity meter.
c. Assorted glassware.

7.7.3.3.2 Procedure

The resistivity cell should be calibrated with known concentrations of sodium chloride solutions, resulting in a graph similar to that shown in Figure 7-3. A portion of the solution obtained by leaching the core is placed in the resistivity cell. The resistivity is measured on a suitable meter and calculated in ohmmeters. With the use of a standard graph (Figure 7-3) and suitable calculations, the resistivity value is converted to a salinity value for the core water. Additional details on apparatus and procedures may be found in ASTM D1125-82, *Method of Test for Electrical Conductivity for Water*.[24,25]

7.7.3.3.3 Precautions

Precautions for this method include:

a. Resistivity meter and cell must be calibrated.
b. Resistivity value must be corrected to a standard temperature.
c. Be certain that electrodes of the resistivity cell are clean before making a measurement.
d. Calibrate electrode with known standard salt solution over the entire range of use to determine/check cell constant as described by Worthington, et al.[25]
e. Hydrogen sulfide may cause some electrodes to change calibration.

7.7.3.3.4 Calculations

Calculations for this method are as follows:

a. The salinity of the core water expelled from the core sample can be calculated directly. The resistivity of the salt solution can be converted directly to concentration milligrams of salt per kg of solution, using a conversion chart of the type illustrated in Figure 7-3.[20]
b. When core water cannot be expelled and the salts are water extracted, the salinity of the originally present core water must be back calculated. The salinity from the resistivity measurement method is converted to total milligrams of sodium chloride leached from the sample. This number represents the total salt from the core water of the sample. The total salt in milligrams and the weight of the volume of core water obtained by saturation tests are used to calculate the concentration of salt in the core water, and this value is expressed in parts per million of sodium chloride. The salinity (mg/kg) of the core water is obtained by following the calculation outlined in 7.7.3.1.3b.

7.7.3.3.5 Advantages

Advantages of this method include:

a. Rapid determination.
b. The resistivity data relate directly to electric log measurements.
c. The total equivalent sodium chloride concentration is determined.

7.7.3.3.6 Limitations

All ions present in the water are calculated as sodium chloride.

7.7.3.3.7 Accuracy/Precision

See 7.7.3.1.6 for general comments and to ASTM D1125[24] for resistivity specific information.

7.7.3.4 Determination of Cations

The direct analysis for sodium, potassium, calcium, and magnesium allow for the estimation of core water salinity and may provide a method to determine if the core water is free of coring fluid. Other cations are present in formation waters but typically account for less than 5 percent of the salinity. Core water salinity can also be estimated from the concentrations of these four cations.

The concentration of these cations can be measured rapidly, simultaneously, and accurately on an inductively coupled plasma atomic emission spectrometer (ICPES). These cations can also by determined individually with an atomic absorption spectrometer (AAS).

Some modern drilling/coring fluids contain high concentrations of potassium. In most formations waters, the ratio of sodium to potassium is much greater than one. Thus for

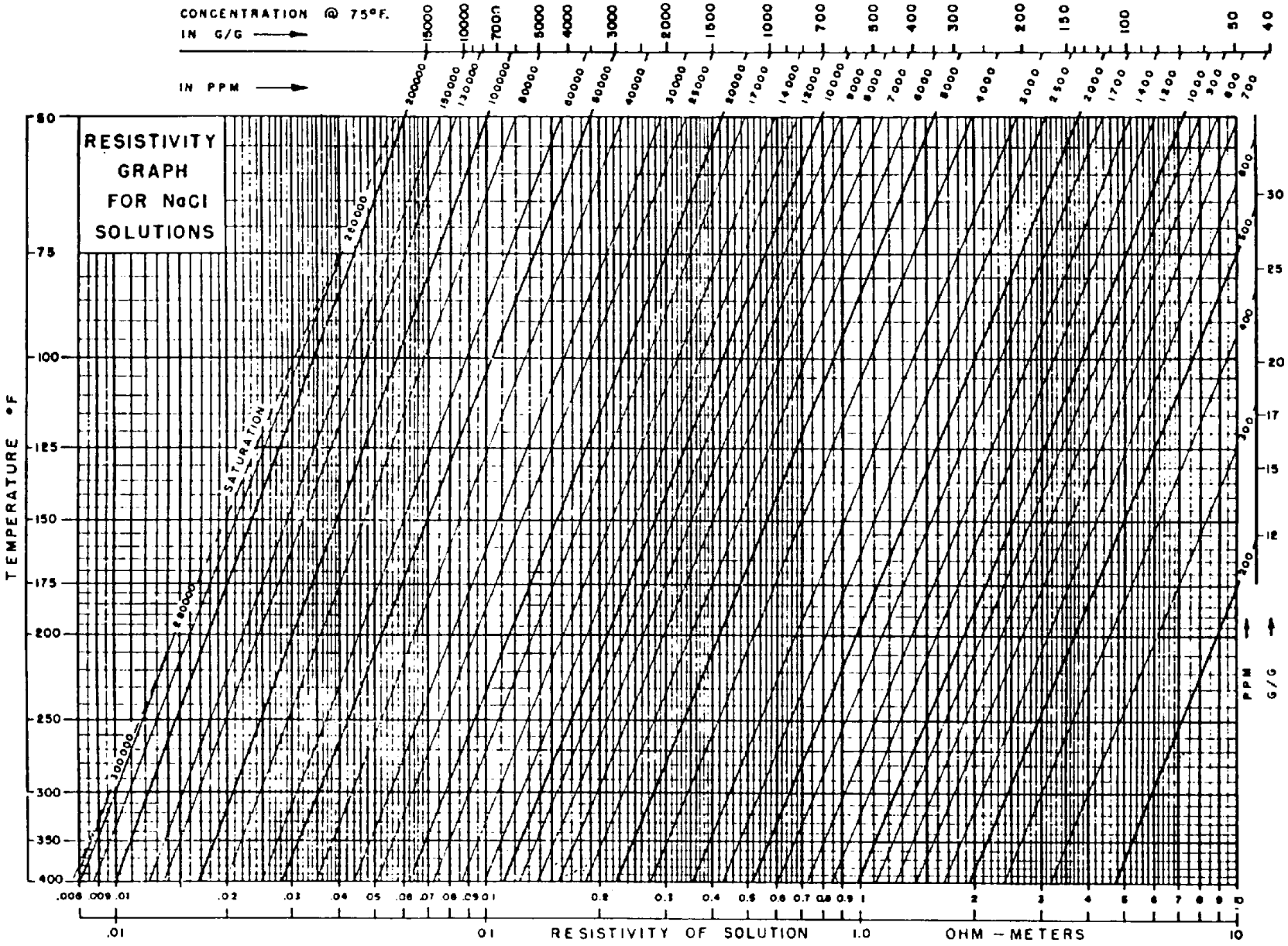


Figure 7-3—Conversion Chart for Salinity Determination Resistivity Versus Temperature and Salinity

potassium-based coring fluids, invasion can be estimated based on the ratio of potassium to sodium in the core water.

7.7.3.4.1 Apparatus and Procedure

The apparatus and procedure for cation determination using AAS are described in detail in ASTM D4191[26] for sodium, ASTM D4192[27] for potassium, and ASTM E508[28] for calcium. The apparatus and procedure for cation determination using ICPES are described in detail in ASTM D1976.[29]

7.7.3.4.2 Precautions

Precautions for this method include:

a. For salinity calculations, the anion is assumed to be chloride even though there are commonly significant sulfate and bicarbonate ions and smaller amounts of other ions.
b. Coring fluid invasion estimation using Na/K ratio requires a knowledge of the interaction of potassium with the sodium bentonite clay in the coring fluid and with the clays in the formation rock.
c. An understanding of local evaporites and their effects on sample preparation is required.
d. The analysis should be performed by personnel trained in these techniques and in a laboratory with the equipment permanently installed.

7.7.3.4.3 Calculations

The methods described in 7.7.3.1.3 for calculating sodium chloride concentration in the undiluted brine and the core leachate can be used to calculate the concentration of cations by replacing the molecular weight of sodium chloride with the atomic weight of the cation.

7.7.3.4.4 Advantages

Advantages of this method include:

a. Both ICPES and AAS are accurate analytical methods.
b. Coring fluid invasion estimates can be made with these data.
c. Cation analysis can provide a more complete picture of the brine salinity instead of assuming that sodium is the only cation present when total salinity is based only on chloride analysis.

7.7.3.4.5 Limitations

The lack of availability and high cost of ICPES and AAS analyses are a limitation.

7.7.3.4.6 Accuracy/Precision

Refer to 7.7.3.1 for general comments and to ASTM D4191, D4192, E508, and D1976 for more specifics.[26-29] In general, AAS and ICPES should be able to determine the concentration of these cations with an accuracy and precision of 1% to 2%.

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SECTION 8—REPORTING

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8 Reporting

8.1 INTRODUCTION

An essential part of a core analysis program is the data documentation phase. Typical data reporting formats are tabular, graphical, and/or digital. These constitute the permanent record(s) of the testing program. Such reports are furnished to the user and are filed by the testing laboratory in accordance with sound archiving procedures.

Tables 8-1 and 8-2 are developed for data documentation at the wellsite and in the laboratory. Commentary which may serve to aid in the understanding and/or the interpretation of the data should be furnished by the way of attachments and/or footnotes. Deviations from accepted standard practices must be noted. Such deviations may include: (a) unusual coring conditions, (b) alterations to the planned coring fluid design, (c) necessary changes in planned on-site or laboratory core handling/preservation methods, and (d) unusual laboratory procedures, etc. The report should contain sufficient wording so as to leave no question or doubt as to the testing methods that were employed.

8.2 TABULAR REPORT

A tabular report should include all of the data, positively identified and tabulated, in some convenient form. Identification should include such items as listed in Tables 8-1 and 8-2. Porosity values should be reported in percent to 0.1 whereas the grain density values should be reported to 0.01 g/cm^3 accuracy. The permeability values should be reported with no less than three significant figures. The specific presentation of data may be determined between the user and the analysts. The suggested numbers of significant figures to be reported are as follows:

Porosity values to	3	(e.g. 0.251 fraction or 25.1%)
Grain density values to	3	(e.g. 2.65 g/cm^3)
Saturation values to	3	(e.g. 50.5% pore volume)
Pore volume values to	4	(e.g. 21.85 cm^3)
Permeability values to	3	(e.g. 2.55 x 10^{+3} md)

8.3 GRAPHICAL REPORT

Graphical presentations are often included to provide the user with a pictorial overview of various core data. Through the continued advances in computer graphic software a large number of pictorial formats are readily available including crossplots, histograms, and core data profiles (logs). The selection of any specific format is left to the discretion of each individual core analyst; however, two graphs have been widely accepted and are recommended for inclusion in every basic core analysis report: a *Permeability vs. Porosity* plot and a *Core Data vs. Depth (sub-surface or sub-sea)* plot.

8.3.1 Permeability vs. Porosity Crossplots

A plot of permeability vs. porosity, with porosity plotted on a linear x-axis and permeability on a logarithmic y-axis, has been included in most conventional core analysis (percussion sidewall data reports rarely contain these crossplots) reports (see Figure 8-1). Also, some best-fit lines are “estimated” while others are derived by curve-fitting procedures. The latter is not always the better approach, depending on the amount of data and the presence of out-liers or fliers that may or may not impact a mathematical approach. A standard labeling procedure should include the essential data identification as adopted through the rest of the core report, as well as any assumptions, notations, or scaling unit parameters unique to each specific plot. While the selection of axis scaling is data dependent and may vary according to the intent of the analyst, a general recommendation is made to select an appropriate scale which will allow the data to fill the entire plot while maintaining easily interpolated axis divisions such as unit divisions of 2, 5, or 10.

8.3.2 Core Data Profiles

The core profile log is a multi-plot figure sometimes referred to as a “core analysis log” (see Figure 8-2). This multi-plot provides a pictorial overview of a variety of core data as a function of sample depth. Generally included on this plot is a gamma ray log, a log of permeability, porosity, reservoir fluids saturation, and possibly a log of bulk or grain density. The recommended standard for vertical scale is 5 inches per 100 feet of depth. The horizontal scale will vary with a log and should be individually selected to allow the data to span the entire plot. A standard labeling procedure should include the essential data identification, as adopted through the rest of the core report, as well as any assumptions, notations, or scaling unit parameters unique to each specific plot.

8.4 DIGITAL REPORT

Current and emerging technologies provide means for core analysis data to be recorded automatically in a digital format. Initial (raw) and calculated (final) data storage, as a result, is being done increasingly on computer systems. Current technology offers a wide choice of data gathering and storage methods. A standardized method of data reporting within the petroleum industry could reduce the cost and effort required to translate data between the various computer platforms.

The Digital Core Analyses Interchange Standard (DCIS) defines the logical data organization on any type of physical

Table 8-1—Core Analysis Wellsite Data

Prepared By: _____ Date: M M D D Y Y Core Number _____

Operator _____ Coring Company _____ Core Analysis Company _____

Contract/Phone _____ Contact/Phone _____

Well Identification _____ Field Reservoir _____ Address _____

API Number _____ ❑ Vertical Well ❑ DeviatedWell ❑ Sidetrack _____

County/State/Country _____ Legal Location/Block _____ Elevations: _____ Ground/Mean Sea Level/ _____ Derrick Floor Kelly Bushing ____

Persons Requesting Work _____ Phone _____ Fax _____ Address _____

Report Results To _____ Phone _____ Fax _____ Address _____

Alternate _____ Phone _____ Fax _____ Address _____

Rig Contract _____ Phone _____ Fax _____ Address _____

Coring/Drilling Fluid
Type/Contents _____ Weight _____ ppg Funnel Viscosity _____ sec Water Loss _____ cm^3/30 min Chlorides _____ ppm pH _____ Tracer _____

Type of Core
❑ Conventional _____ Inner Barrel Type _____
❑ Sponge ❑ Oriented Barrel
❑ Pressure Retained ❑ Other _____
Sidewall: ❑ Percussion or ❑ Mechanically Drilled Number Attempted _____ Number Recovered _____

Analysis Planned
❑ Plug Size or ❑ Full Diameter
Method
❑ Fluid Saturation _____
❑ Porosity _____
❑ Grain Density _____
❑ Permeability _____
❑ Surface Gamma Log _____
❑ Special Instructions _____

Transportation
❑ By Service Company _____
❑ Ground _____
❑ Air _____
❑ Other _____
Carrier _____
Date Shipped _____

Preservation
Method ❑ Plastic Laminate/Type _____
❑ Freeze ❑ Dry Ice or ❑ Liquid Nitrogen
❑ Refrigerate _____ °F _____°C
❑ Core Inner Barrel
❑ Core Wrap and/or ❑ Dip-Type ❑ Resination
❑ Other

Exposure Time/Climate _____
Notes _____

Laboratory/Long Term Preservation _____
Special Instructions _____
Post Core Analysis Instructions/Core Material Distribution _____

Formation(s)/ Zones _____
Expected Core Point _____
_____
Expected Core Point _____
_____
Expected Core Point _____

Depths (Drillers)
Interval Cut _____
Interval Recovered _____
Interval Cut _____
Interval Recovered _____
Interval Cut _____
Interval Recovered _____

Well Inclination _____ Core Diameter _____ ❑ In ❑ cm
Total Core Recovered _____ Bit Type _____
Length Cored _____ ❑ ft ❑ m Percent Recovery _____
Coring Time _____ Trip Time _____
Estimated Connate Water Salinity _____ ppm Chlorides or Estimated R_W _____ @ _____ ❑ °F ❑ °C
Estimated Production ❑ Oil _____ °API
❑ Condensate
❑ Dry Gas

Notes _____

Attach Coring Log and Core Description

Table 8-2—Basic Core Analysis Laboratory Data

General Information
Analysis Requested Date M M D D Y Y API # _____ File # _____

Requested By: _____

❑ Full Diameter or ❑ Plug : Size _____ ❑ In ❑ cm

Measurement Method/Special Instructions

❑ Saturations (O,W,G) _____

❑ Porosity _____

❑ Permeability _____

❑ Grain Density _____

❑ Other _____

Objective of the tests as agreed by parties involved _____

_____

Transportation and Inventory

Carrier _____ Date Shipped _____ Core Arrival Date _____

Core condition on arrival:

❑ Preserved ❑ Frozen ❑ Unpreserved ❑ Cleaned ❑ Other _____

Correlation Depths: Driller's _____ Log _____ TVD _____ ❑ ft ❑ m

Allocation of the missing core dinterval: ❑ Bottom ❑ Middle ❑ Top _____

Screening

❑ CT ❑ X-ray ❑ NMR ❑ Fluroscopy ❑ Other _____

Sample Handling

Core Gramma (yes/no, type) _____

Lab Preservation (prior to analyses) _____

Sampling Method

Sample Sleeve ❑ Yes ❑ No, Type _____

Nominal Sample Diameter/Length_____ ❑ In ❑ cm

Plugging/Trimming Fluid_____

Treatment prior to testing (flushing, evaluation, resaluration, etc.) _____

_____

Core Disposition (removed samples, storage location, preservation, etc.)_____

_____

Lab Analyst _____

Other Service

❑ Slab ❑ Resination ❑ Photo: ❑ BW ❑ Color ❑ U.V. ❑ Other _____

Supplementary Tests _____

_____

Methods and Conditions

Cleaning:

Methods	Conditions
❑ No Cleaning	Solvents _____
❑ Dean-Stark	Temperature _____ ❑ °F ❑ °C
❑ Soxhlet	Pressure _____ ❑ psi ❑ kPa
❑ CO2/Solvent	Time _____
❑ Flow Through	Volume and Rate_____ ❑ cc ❑ cc/sec
❑ Others	Others _____

Drying:

Methods	Conditions
❑ Convection oven	Temperature _____ ❑ °F ❑ °C
❑ Vacuum oven	Time _____
❑ Humidity oven	Relative Humidity % _____
❑ Others	

Porosity: Methods

Pore Volume	Grain Volume	Bulk Volume
❑ Boyle's Law	❑ Boyle's Law	❑ Caliper
❑ Saturation	❑ Archimedes	❑ Archimedes
❑ Summation-Of-Fluids	❑ BV-GV	❑ Mercury Displacement
❑ BV-GV	❑ Other	❑ GV+PV
❑ Other		❑ Other

Conditions/Fluids

Confining Stress (Magnitude and Type)_____ ❑ psi ❑ kPa

Gas _____ Liquid_____

Pressure _____

Permeability:

Methods	Conditions
❑ Steady State	Fluid Type _____
❑ Unsteady State	Confining Stress _____ ❑ psi ❑ kPa
❑ Probe	Sleeve Durometer _____
❑ Empirical	Pore Pressure _____Units _____
❑ Not Measured	Klinkenberg: ❑ Measured ❑ Empirical ❑ No Correction
❑ Others	Inertial Factor: ❑ Measured ❑ Empirical ❑ No Correction

Saturation:

Methods	Conditions
❑ Distillation Extraction (DS)	Temperature _____ ❑ °F ❑ °C
❑ High Temperature Retort	Fluids _____
❑ Others	Water Density_____ ❑ g/cm³ ❑ kg/m³
	Oil Density _____ ❑ g/cm³ ❑ kg/m³
Correction for Salt:	❑ Corrected for Water Volume
	❑ Corrected for Oil Weight ❑ Not Corrected

Quality Assurance: (Page number in report where the information is provided) _____

_____

Comments/Remarks: _____

Data Anomalies: _____

Client Company:		Date:
API Number:		Other Information:
Well Identification:		
Legal Location/Block:		
Formation:		
Interval Analyzed:		
County/State/Country:		
Field/Reservoir:		

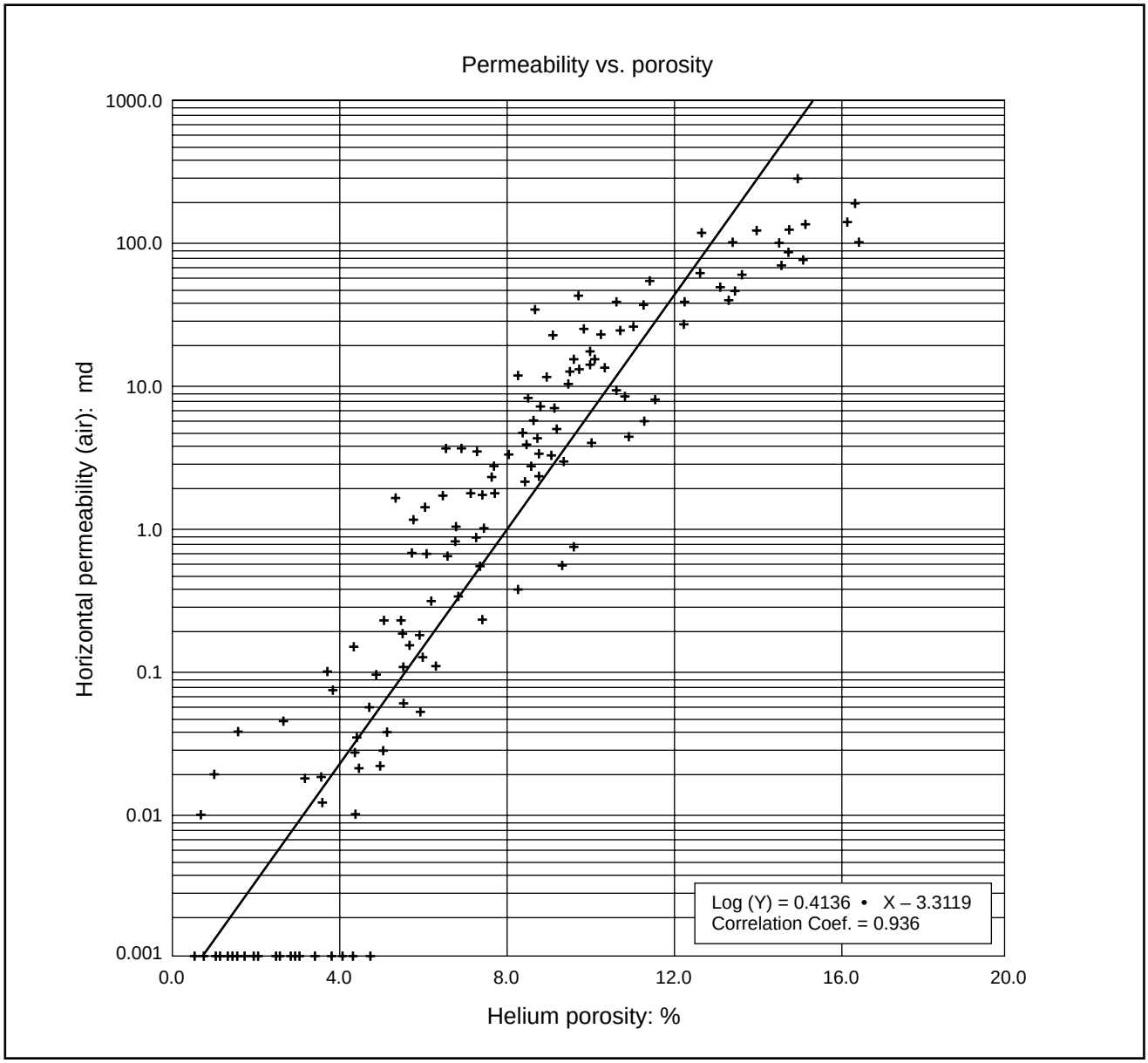

Figure 8-1—Permeability vs. Porosity

Client Company:		Date:
API Number:		Other Information:
Well Identification:		
Legal Location/Block:		
Formation:		
Interval Analyzed:		
County/State/Country:		
Field/Reservoir:		

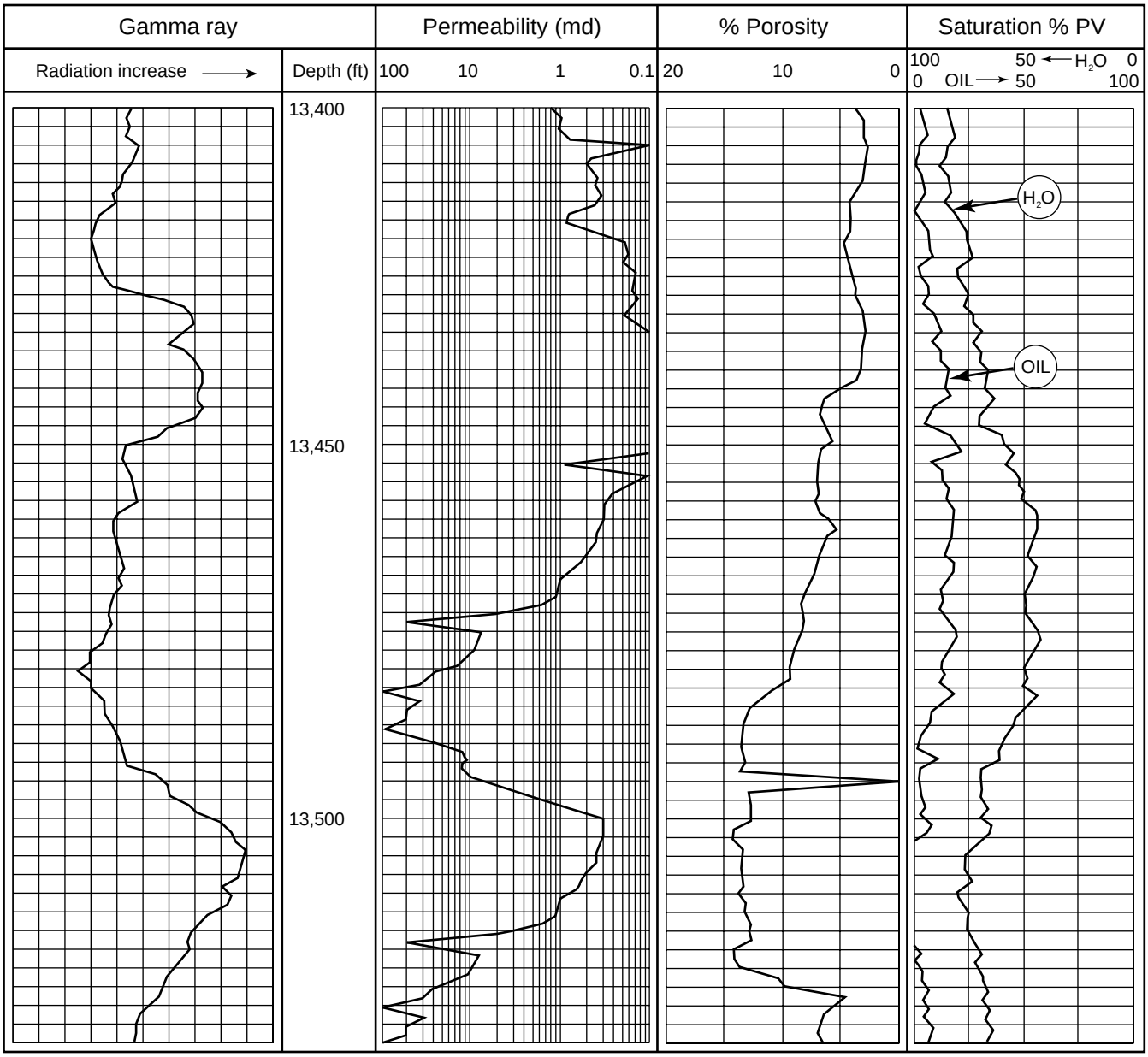

Figure 8-2—Core Data Profile

storage media (i.e., floppy disk, magnetic tape, optical disk, etc.). The physical format is the way in which stored data is located and organized on a physical medium. The specific way in which the logical and physical formats are bound depends on the medium and access mechanism and therefore are beyond the scope of this standard.

8.4.1 Digital Format

The DCIS format consists of the following elements:

a. Logical Record—A group of 8-bit bytes or data characters.
b. Logical File—A group of related Logical Records.

At the byte level, DCIS data are an ordered stream of 8-bit bytes, in which byte k precedes byte k+1. Data encryption is not allowed. No high-order bytes are allowed; all data should be represented in printable ASCII characters (ASCII 32 through ASCII 126). The only exception is ASCII 13 (carriage return) and ASCII 10 (line feed) at the end of each logical record. A combination carriage return and line feed signifies the end of the Logical Record. All records start at the byte location immediately following the carriage return-line feed combination of the previous record.

The layout of the logical file should be as follows (see example in Figure 8-3).

8.4.1.1 Logical File Header

This segment provides general information about the rest of the Logical File. Each record in this segment, as in other segments, is terminated by a carriage return, followed by a line feed character. Starting at logical location of byte 1 at record number 1, the following information should be present:

a. Record 1—Record Count: A positive integer, represented as ASCII characters, right justified, with no embedded commas, and with preceding blanks, if needed, to define the number of logical records including the record count in this logical file. This number includes records in the Logical File Header.
b. Record 2—Maximum Record Length: A non-negative integer represented as 3 ASCII characters, right justified, and with preceding blanks, if needed. A value of 0 indicates that the maximum length of a record is unknown. A positive value indicates that no record in the Logical File will exceed Maximum Record Length in size. A positive value need not correspond to the actual maximum record length; it needs only be an upper bound.

8.4.1.2 Logical File Body Header

The digital presentation of Basic Core Analysis reports should closely follow the format and layout of the hard copies. A maximum of 15 records is allowed in this section. Each record in this section is represented as ASCII characters, left justified, with trailing blanks (designated by the ASCII space character) allowed. The only exception to this is the first record in this segment, the “Number of Records In Logical Body Header,” which is represented by a two character, right justified, positive integer. The Logical File Body Header should consist of the following, as a minimum, in this order:

a. Number Of Records In Logical File Body Header (this is included in the count).
b. Company.
c. Well.
d. Field.
e. Formation.
f. State.
g. County.
h. Core Type.
i. Mud Type.
j. Elevation (KB).
k. Date Of Report.
l. Analyzed by.

The first characters in each record in this segment must define the record (e.g. Company, Well, Field, etc.). A colon, :, is used as a separator between the definition and the value.

8.4.1.3 Logical File Body

This segment contains the main body of the core analyses data. One record is devoted to each sample reported in the hard copy report. This segment consists of:

a. Column Header—This is a segment giving the layout of the information to follow in the next segment. Information in this segment will provide the definitions for each column of data. A maximum of 25 records is allowed in this section. Each record in this section is represented by ASCII characters, left justified, with trailing blanks (designated by the ASCII space character) allowed. The only exception to this is the first record in this segment, the “Number Of Records In Logical File Body Column Header,” which is represented by a two character, right justified, positive integer. It should be remembered that the count of records, identified as the “Number of Records in Logical File Body Header” found within the Logical File Body Header includes the line listing of the column header record count. This segment should contain, as a minimum, the following:

1. Number Of Records In Logical File Body Column Header (including this one in the count).
2. Sample Number.
3. Depth.
4. Sample type (plug, full diameter, etc.).

Additional records in this segment must specify the layout of additional data columns (e.g. permeability, oil saturation, etc.). Units of measurement must be included (%PV, gram, cm^3, etc.).

b. Body—This segment contains the main body of core analysis data, the layout of which is defined by the preceding segment. Various data columns for each sample analyzed must be separated by at least two ASCII space characters. The layout should be the same as that for the hardcopy of the report.

8.5 QUALITY ASSURANCE

Quality assurance/quality control (QA/QC) is an integral component of the core analysis process. This component is the foundation of all laboratory techniques and management. QA/QC impacts all technical decisions, training of personnel, selection of personnel and analytical equipment, use of instruments and equipment, and adherence to defined standards and quality control methodologies. In addition, operational procedures, i.e., regular procedural audits, documentation control, and quality control verifications are also impacted. QA/QC should be considered in the planning phase, before actually cutting the core. Quality assurance is the examination of the overall process and procedures used to obtain the data. Cost restraints and the purpose or use of the data may govern the testing methods and thoroughness used in obtaining data. How well the data represent the in situ formation may be quite uncertain and its assessment should be left to the evaluating engineer and/or the end user.

Quality control is the process of evaluating a particular testing method or instrument. It is a procedure which results in definable error limits as determined from the current study, or in a statement of precision which the laboratory has established based on comparable measurements of standard samples.

Quality control data should be supplied with every direct or indirect measurement along with the measurement procedures to provide the beginning of quality assurance. Quality control is the responsibility of the laboratory; and quality assurance is the responsibility of both the laboratory and the end user.

Proprietary procedures need not be divulged in detail in the report.

A high standard of QA/QC depends upon commitment by analysts, supervisor, laboratory director, and QA manager to quality production in strict accordance to established protocols. The International Organization for Standardization and National Measurement Accreditation Service are two organizations that are dedicated to setting quality assurance standards.

29	Record Count							Logical File Header
225	Maximum Record Length							
16	No. of Records in Logical File Body Header							
Company:	Gusher Oil Company							
Well:	Gusher #1							
API #:	3507123474							
Section:	18							
Township:	T-42-S							
Range:	R-18-E							
Field:	Gusher							Logical File Body Header
Formation:	Huge							
State:	California							
County:	Los Angeles							
Core Type:	Conventional With plastic Liner							
Mud Type:	Water base							
Elevation (KB):	20 ft. (6.1 meters)							
Date of Report:	June 9, 1992							
Analyzed by:	M. M. H.							
9	No. of Records in Logical File Body Column Header							
Sample Number								
Depth (ft.)								
Sample type								
Permeability (md)								Logical File Column Header
Porosity (% BV)								
Oil Saturation (% PV)								
Water Saturation (% PV)								
Lithology								
1	1234	“Whole Core”,	12.3	35.5	30.0	42.0	SS: 1t brn, slt-med gr, p cmt	Body
2	1235	or “Plug”, or	10.3	30.2	29.0	50.8	SS: 1t brn, slt-med gr, p cmt	
3	1236	“Sidewall, etc.	0.3	8.5	21.3	62.3	SS: 1t brn, slt-med gr, p cmt	

Figure 8-3—Example of DCIS Logical File

8.6 QA/QC PROTOCOL

8.6.1 Step 1

a. Ensure that the Standard Operating Procedure (SOP) is compatible with sample type and supervisor's instructions.

b. Assume custody of all or portions of the samples.

c. Set up instrument/apparatus consistent with specified SOP.

d. Perform analysis (including calibration) according to specified SOP.

e. Derive data from analyses and ensure that data is within control limits.

f. Maintain all results, instrument control, calibration checks, and documentation in accordance with SOP documentation requirements.

g. Maintain all QA/QC data control charts.

8.6.2 Step 2

a. Review procedural requirement for sample type and ensure that correct SOP was followed.

b. Review all data generated and check calculations.

c. Review QA/QC criteria and ascertain that the data are within limits.

d. Evaluate results for reanalysis if data are not within QA/QC limits and note in report when acceptance limits have been exceeded.

e. Prepare a final report and submit it to the Laboratory Director.

8.6.3 Step 3

a. Review final report data and compare each new set of data with previously completed data for consistency.

b. Verify that the data are within acceptable QA/QC criteria limits.

c. Order reanalysis of sample when necessary and if possible.

d. Review final report for analytical quality, custodial documentation, and completeness. Confirm that all laboratory procedures and documentation satisfy in-house and client requirements.

8.6.4 Step 4

a. Perform completeness audits of randomly and non-randomly selected final reports. This audit includes document verification, data reduction, and report narrative evaluations.

b. Review final reports for analytical and documentary completeness, client or contractual requirements, and timeliness.

c. Report to senior management.

d. Perform biannual system audits with blind check samples and periodic performance evaluation studies.

Tabulated data in written reports could follow the format presented in Tables 8-3 through 8-8. Standard abbreviations for lithologic descriptions could follow the nomenclature presented in Tables 8-9 and 8-10. Table 8-11 presents some of the SI units, conversion factors, and equations that are often used by core analysts. Table 8-12 presents common nomenclature.

Table 8-3—Basic Core Plug Analysis Report*

Client Company:		Date:
API Number:		Other Information:
Well Identification:		
Legal Location/Block		
Formation:		
Interval Analyzed:		
County/State/Country:		
Field/Reservoir:		

Sample Number	Depth[a] ft. or m	Interval represented, ft. or m	Gas Permeability[b], md k_h $k_{h\infty}$[c] k_v $k_{v\infty}$[c]	Pore Volume[d] cm^3	Porosity[b] % BV	Grain Density g/cm^3	Saturation, % PV Oil Water Gas	Lithology[e]
_____								
_____								
_____								

*This form can also be used for reporting core analysis of rotary sidewall plugs.
[a]The indicated depth is driller's depth to tenth of the foot or hundredth of meter (0.1 ft. or 0.01 m).
[b]The confining stress should be specified. See Table 8-2 for amount and type of stress used during measurements.
[c]Whether the correction for gas slippage or inertia is made (extrapolation or correlation) and the type of gas used for flow should be specified.
[d]Optional, intended for data QA/QC.
[e]See the standard abbreviation table.

Table 8-4—Basic Full Diameter Core Analysis Report

Client Company:		Date:
API Number:		Other Information:
Well Identification:		
Legal Location/Block		
Formation:		
Interval Analyzed:		
County/State/Country:		
Field/Reservoir:		

Sample Number	Depth[a] ft. or m	Gas Permeability[b], md k_{max}[c] k_{90}[c] k_v[c]	Pore Volume[d] cm^3	Porosity[b] % BV	Grain Density g/cm^3	Saturation, % PV Oil Water Gas	Lithology[e]
_____							
_____							
_____							

[a]In full diameter core analysis, the depth associated with the actual top and bottom of the sample should be specified.
The indicated depth should be drillers depth to tenth of the foot or hundredth of meter (0.1 ft. or 0.01 m).
[b]The confining stress should be specified. See Table 8-2 for amount and type of stress used during measurements.
[c]Whether the correction for gas slippage or inertia is made and the type of gas used for flow should be specified (See Table 8-2).
k_{max} is not the maximum permeability but the greater of two measured horizontal permeability values, whereas k_{90} is the horizontal value 90° to k_{max}.
[d]Optional, intended for data QA/QC.
[e]See the standard abbreviation table.

Table 8-5—Basic Sidewall Core Analysis Report

Client Company:		Date:
API Number:		Other Information:
Well Identification:		
Legal Location/Block		
Formation:		
Interval Analyzed:		
County/State/Country:		
Field/Reservoir:		

Recovered length in. or cm	Depth[a] ft. or m	Permeability, md (empirical)	Porosity % BV	Pore Volume[b] cm^3	Saturation, % PV (retort) Oil Water	Saturation[c] % PV Oil Gas	Probable[d] Production	Critical Water[e] Saturation, % PV (empirical)	Gas[f] Detector Units	Lithology[g]
_____										
_____										
_____										

[a]The indicated depth is the wireline depth to tenth of the foot or hundredth of meter (0.1 ft. or 0.01 m).
[b]Optional, intended for QA/QC.
[c]Optional.
[d]Optional, expected fluid to be produced.
[e]Optional, estimated reservoir water saturation above which one should expect water production (empirical value obtained from correlation).
[f]Presence of hydrocarbon gas measured by conductivity of hot wire element inserted into each sidewall jar or suitable container used to transport each core.
[g]See the standard abbreviation table.

Table 8-6—Basic Oil-Wet Sponge Core Analysis Report

Client Company:		Date:
API Number:		Other Information: Oil density used in calculations, _____ ❑g/cm³ ❑kg/m³
Well Identification:		
Legal Location/Block		
Formation:		
Interval Analyzed:		
County/State/Country:		
Field/Reservoir:		

Sample Number	Depth[a] ft. or m	Permeability[b], md k_{max}[c] k_{90}[c] k_v[c]	Pore Volume[b] cm³	Porosity % BV	Grain Density g/cm³	Sponge Oil Saturation %PV[e]	Core Oil Saturation %PV	Total Oil Saturation %PV (Core + Sponge)	Water Saturation %PV	Lithology[f]
_____										
_____										
_____										

[a]In full diameter core analysis, the depth associated with the actual top and bottom of the sample should be specified. The indicated depth should be drillers depth to tenth of a foot or hundredth of a meter (0.1 ft. or 0.01 m).
[b]The confining stress should be specified. See Table 8-2 for amount and type of stress used during measurements.
[c]Whether the correction for gas slippage or inertia is made and the type of gas used for flow should be specified (See Table 8-2). k_{max} is not the maximum horizontal permeability value, whereas k_{90} is the horizontal value 90° to k_{max}.
[d]Optional, intended for data QA/QC.
[e]Standard condition should be specified.
[f]See the standard abbreviation table.

Table 8-7—Basic Full Diameter Pressure-Retained Core Analysis Report

Client Company:		Date:
API Number:		Other Information:
Well Identification:		
Legal Location/Block		
Formation:		
Interval Analyzed:		
County/State/Country:		
Field/Reservoir:		

Sample Number	Depth[a] ft. or m	Gas Permeability[b], md k_{max}[c] k_{90}[c] k_v[c]	Pore Volume[d] cm³	Porosity[b] % PV	Grain Density g/cm³	Saturation, % PV Pressure Depletion Oil Water	Saturation, % PV Total @ Stand. Cond.[e] Oil Water	Lithology[f]
_____								
_____								
_____								

[a]In full diameter core analysis, the depth associated with the actual top and bottom of the sample should be specified. The indicated depth should be drillers depth to tenth of a foot or hundredth of a meter (0.1 ft. or 0.01 m).
[b]The confining stress should be specified. See Table 8-2 for amount and type of stress used during measurements.
[c]Whether the correction for gas slippage or inertia is made and the type of gas used for flow should be specified (See Table 8-2). k_{max} is not the maximum permeability but the greater of two measured horizontal permeability values, whereas k_{90} is the horizontal value 90° to k_{max}.
[d]Optional, intended for data QA/QC.
[e]Standard condition should be specified.
[f]See the standard abbreviation table.

Table 8-8—Basic Core Fracture Analysis Report

Client Company:		Date:
API Number:		Other Information:
Well Identification:		
Legal Location/Block		
Formation:		
Interval Analyzed:		
County/State/Country:		
Field/Reservoir:		

Sample Number	Depth[a] ft. or m	Gas Permeability[b], md k_h, N-S[c] k_h, E-W[c] k_h, NW-SE[c] k_h, NE-SW[c]	Gas Permeability[b], md k_v[c]	Pore Volume[d] cm^3	Porosity[b] % BV	Grain Density g/cm^3	Saturation, % PV Oil Water	Lithology[e]
_____								
_____								
_____								

[a]In full diameter core analysis, the depth associated with the actual top and bottom of the sample should be specified. The indicated depth should be drillers depth to tenth of a foot or hundredth of meter (0.1 ft. or 0.01 m).
[b]The confining stress should be specified. See Table 8-2 for amount and type of stress used during measurements.
[c]Whether the correction for gas slippage or inertia is made and the type of gas used for flow should be specified.
[d]Optional, intended for QA/QC.
[e]See the standard abbreviation table.

Table 8-9—Standard Abbreviations for Lithologic Descriptions (Sorted alphabetically by term.)

about	abt
above	abv
absent	abs
abundant	abd
acicular	acic
after	aft
agglomerate	aglm
aggregate	agg
algae algal	alg
allochem	allo
alter (ed ing)	alt
altered altering	alt
amber	amb
ammonite	amm
amount	amt
and	&
angular	ang
anhedral	ahd
anhydrite (ic)	anhy
aphanitic	aph
apparent	apr
appears	aprs
approximate (ly)	aprox
aragonite	arag
arenaceous	aren
argillaceous	arg
argillite	argl
arkose (ic)	ark
as above	a.a
asphalt (ic)	asph
assemblage	assem
associated	assoc
at	@
authigenic	authg
average	av
band (ed)	bnd
Barite (ic)	bar
basalt (ic)	bas
basement	bm
become (ing)	bcm
bed (ded ing)	bdg
bentonite (ic)	bent
bioclastic	biocl
bioherm (al)	bioh
biomicrite	biomi
biosparite	biosp
biostrom (al)	biost
biotite	biot
bioturbated	biotb
birdseye	bdeye
birefringence	bifg
bitumen (inous)	bit
black (ish)	blk blksh
blade (ed)	bld
block (y)	blky
blue (ish)	bl blsh
bored (ing)	bor
botryoid (al)	btry
bottom	btm
boudinage	boudg
boulder	bldr
boundstone	bdst
brachiopod	brac
brackish	brak
branching	brhg
break	brk
breccia (ted)	brec
bright	brt
brittle	brit
brown	brn
bryozoa	Bry
bubble	bubl
buff	bu
Bulbous	bulb
burrow (ed)	bur
calcarenite	clcar
calcareous	calc
calcilutite	clclt
calcirudite	clcrd
calcisiltite	clslt
calcisphere	clcsp
calcite filled fractures	ccf
calcite (ic)	$CaCO_3$
caliche	cche
carbonate	CO_3
carbonized	cb
cavern (ous)	cav
caving	cvg
cement (ed ing)	cmt
center centered	cntr
cephalopod	ceph
chalcedony (ic)	chal
chalk (y)	chk chky
charophyte	char
chert (y)	cht chty
chitin (ous)	chit
chitinozoa	chtz
chlorite (ic)	chlor
chocolate	choc
circulate (ion)	circ
clastic	clas
clay (ey)	cly
claystone	clyst
clean	cln
clear	clr
cleavage	clvg
cluster	clus
coal	coal

(continues)

Table 8-9—(Continued)

coarse (ly ness) ..... crs
coated grains ..... ctd gn
coated (ing) ..... ctd
cobble ..... cbl
color (ed) ..... col
common ..... com
compact ..... cpct
compare ..... cf
concentric ..... cncn
conchoidal ..... conch
concretion (ary) ..... conc
conglomerate (ic) ..... cgl
conodont ..... cono
considerable ..... cons
consolidated ..... consol
conspicuous ..... conspic
contact ..... ctc
contamination (ed) ..... contm
content ..... cont
contorted ..... cntrt
coquina (oid) ..... coq
coral coralline ..... cor corin
core c
covered ..... cov
cream ..... crm
crenulated ..... cren
crevice ..... crev
crinkled ..... crnk
crinoid (al) ..... crin crinal
cross x
crossbedded (ing) ..... xbd xbdg
crosslaminated ..... xlam
crossstratified ..... xstrat
crumpled ..... crpld
cryptocrystalline ..... crpxl
cryptograined ..... crpgr
crystal (line) ..... xl xln
cube (ic) ..... cub
cuttings ..... ctgs
dark (er) ..... dk dkr
dead ..... dd
debris ..... deb
decrease (ing) ..... decr
dendritic ..... dend
dense (er) ..... dns
depauperate ..... depau
description ..... descr
desiccation ..... dess
determine ..... dtrm
detrital (us) ..... dtrl
devitrified ..... devit
diabase ..... db
diagenesis (etic) ..... diagn
diameter ..... dia
difference ..... dif
disseminated ..... dism
distillate ..... dist
ditto ..... “
dolocast (tic) ..... dolc
dolomite (ic) ..... dol
dolomold (ic) ..... dolmd
dolostone ..... dolst
dominant (ly) ..... dom
drill stem test ..... DST
drilling ..... drlg
druse ..... dru
drusy ..... drsy
earthy ..... ea
east ..... E
echinoid ..... ech
elevation ..... elev
elliptical ..... elip
elongate ..... elg
embedded ..... embd
enlarged ..... enl
equant ..... eqnt
equivalent ..... equiv
euhedral ..... euhd
euxinic ..... eux
evaporite (itic) ..... evap
excellent ..... ex
expose (ed ure) ..... exp
extraclast (ic) ..... exclas
extremely ..... extr
extrusive rock, extrusive (ion) ..... extr
facet (ed) ..... fac
faint (ly) ..... fnt
fair ..... fr
fault (ed) ..... flt
fauna ..... fau
feet ..... ft
feldspar (athic) ..... fspr
fenestra (al) ..... fen
Ferromagnesian ..... Femag
ferruginous ..... Fe
fibrous ..... fibr
figured ..... fig
fine (ly) ..... f fnly
fissile ..... fis
fissures ..... fiss
flaggy ..... flg
flake (s y) ..... flk flks flky
flat ..... fl
flesh ..... fls
floating ..... fltg
flora ..... flo
fluorescence (ent) ..... flor
foliated ..... fol
foot ..... ft
foraminifera (al) ..... foram

(continues)

Table 8-9—(Continued)

formation....fm
fossil (iferous)....fos
fracture (d)....frac
fragment (al)....frag
framework....frmwk
frequent....freq
fresh....frs
friable....fri
fringe (ing)....frg
frosted quartz grains....F.Q.G.
frosted....fros
fucoid (al)....fuc
fusulinid....fus
gabbro....gab
gas....G
gastropod....gast
generally....gen
geopetal....gept
gilsonite....gil
glass (y)....glas
glauconite (itic)....glau
Globigerina (inal)....glob
gloss (y)....glos
gneiss (ic)....gns
good....gd
grade (s ing d)....grd
grain (s, ed)....gr
grainstone....grst
granite wash....grnt.w
granite (ic)....grnt
granule (ar)....grnl
grapestone....grapst
graptolite....grap
gravel....grv
gray, grey (ish)....gry
grysh, graywacke....gwke
greasy....gsy
green (ish)....gn gnsh
grit (ty)....gt
gypsum (iferous)....gyp
hackly....hky
halite (iferous)....hal
hard....hd
heavy....hvy
hematite (ic)....hem
heterogeneous....hetr
heterostegina....het
hexagonal....hex
high (ly)....hi
homogeneous....hom
horizontal....hztl
hornblend....hornbl
hydrocarbon....hydc
igneous rock, igneous....ig
impression....imp
in part....I.P.
inch....in
inclusion (ded)....incl
increasing....incr
indistinct....indst
indurated....ind
inoceramus....inoc
insoluble....insl
interbedded....intbd
intercalated....intercal
intercrystalline....intxl
interfingered....intfr
interfragmental....intfrag
intergranular....intgran
intergrown....intgn
interlaminated....intlam
interparticle....intpar
interpretation....intpt
intersticies (iitial)....intst
interval....intvl
intraclast (ic)....intclas
intraformational....intfm
intraparticle....intrapar
intrusive rock, intrusive....intr
invertebrate....invtb
iridescent....irid
iron....Fe
ironstone....Fest
irregular (ly)....irr
irridescent....irid
isopachous....iso
jasper (oid)....jasp
joint (s, ed, ing)....jt jts jtd
kaolin (itic)....kao
lacustrine....lac
lamina (tions, ated)....lam
large larger....lge
laterite (itic)....lat
lavender....lav
layer....lyr
leached....lchd
ledge....ldg
lens, lenticular....len lent
light (er)....lt
lignite (itic)....lig
limestone....ls
limonite (itic)....lim
limy....lmy
lithic....lit
lithographic....lithgr
lithology (ic)....lith
little....ltl
littoral....litt
local....loc
long....lg

(continues)

Table 8-9—(Continued)

loose	lse
lower	low
lumpy	lmpy
lustre	lustr
lutite	lut
macrofossil	macrofos
magnetite, magnetic	mag
manganese, manganiferous	mn
marble	mbl
marine	marn
marl (y)	mrl mrly
marlstone	mrlst
maroon	mar
massive	mass
material matter	mat
matrix	mtrx
maximum	max
median	mdn
medium	med
member	mbr
meniscus	men
metamorphic (osed)	meta metaph
metasomatic	msm
mica (ceous)	mic
micrite (ic)	micr
microcrystalline	micxl
microfossil (iferous)	microfos
micrograined	micgr
micromicaceous	micmica
microoolite	microol
micropore (osity)	micropor
microspar	microspr
microstylolite	microstyl
middle	mid
miliolid	milid
milky	mky
mineral (ized)	mnrl
minimum	min
minor	mnr
minute	mnut
moderate	mod
mold (ic)	mol
mollusc (a)	moll
mosaic	mos
mottled (ing)	mot
mud (dy)	md mdy
mudstone	mdst
muscovite	musc
nacreous	nac
no show	n/s
no	n
no sample	n.s.
no visible porosity	n.v.p.
nodule (s, ar)	nod
north	N
novaculite	novac
numerous	num
object	obj
occasional	occ
ochre	och
odor	od
oil source rock	OSR
oil	O
olive	olv
Olivine	olvn
oncolite (oidal)	onc
ooid (al)	oo
oolicast (ic)	ooc
oolite (itic)	ool
oomold (ic)	oomol
opaque	op
orange (ish)	orng
orbitolina	orbit
organic	org
orthoclase	orth
orthoquartzite	otz
ostracod	ostr
overgrowth	ovgth
oxidized	ox
oyster	oyst
packstone	pkst
paper (y)	pap
part (ly)	pt
particle	par
parting	ptg
parts per million	ppm
patch (y, es)	ptch
pearl (y)	prly
pebble (ly, s)	pbl
pelecypod	plcy
pellet (al, oids)	pel
pelletoid (al)	peld
pendular (ous)	pend
permeability (able)	perm K k
petroleum petroliferous	pet
Phenocrysts	phen
phlogopite	phlog
phosphate (atic)	phos
phreatic	phr
phyllite, phyllitic	phly
pink	pk
pinkish	pkish
pinpoint porosity	p.p.
pisoid (al)	piso
pisolite pisolitic	pisol
pitted	pit
plagioclase	plag
plant	plt
plastic	plas
platy	plty

(continues)

Table 8-9—(Continued)

polish polished ..... pol
pollen ..... poln
polygonal ..... poly
poor (ly) ..... p
porahyry ..... prphy
porcelaneous ..... porcel
porosity porous ..... por ϕ
possible (ly) ..... pos
predominant (ly) ..... pred
preserved (ation) ..... pres
pressure deformation ..... p d
primary ..... prim
prism (atic) ..... pris
probable (ly) ..... prob
production ..... prod
prominent (ly) ..... prom
pseudo ..... psdo
pseudo oolite (ic) ..... psool
pumicestone ..... pst
purple ..... purp
pyrite (itized itic) ..... pyr
pyrobitumen ..... pybit
pyroclastic ..... pyrcl
pyroxene ..... pyrxn
quartz (ose, ic) ..... qtz qtzs qtzt
radial (ate, ating) ..... rad
radiaxial ..... radax
range (ing) ..... rng
rare ..... rr
recemented ..... recem
recovery (ered) ..... rec
recrystal (lize, ed, ation) ..... rexl rexlzd rexlt
red (ish) ..... rd rdsh
reef (oid) ..... rf
regular ..... reg
remains, remnant ..... rmn
replaced (ment, ing) ..... rep
residue (ual) ..... res
resinous ..... rsns
rhomb (ic) ..... rhb
ripple ..... rpl
rock ..... rk
round (ed) ..... rnd
rounded frosted pitted ..... r.f.p.
rubble (bly) ..... rbl rbly
rudist ..... rud
rugose, ruga ..... rug
rugose coral rugosa ..... rugc
saccharoidal ..... sacc
salt and pepper ..... s & p
salt water ..... s.w.
salt (y) ..... sa
saltcast (ic) ..... sac
same as above ..... a.a.
sample ..... spl
sand (y) ..... sd sdy
sandstone ..... ss
saturation (ated) ..... sat
scales ..... sc
scaphopod ..... scaph
scarce ..... scs
scatter (ed) ..... scat
schist (ose) ..... sch
scolecodont ..... scol
secondary ..... sec
sediment (ary) ..... sed
selenite ..... sel
septate ..... sept
shadow ..... shad
shale (ly) ..... sh shy
shell ..... shl
shelter porosity ..... shlt por
show ..... shw
siderite (itic) ..... sid
sidewall core ..... S.W.C.
silica (iceous) ..... sil
silky ..... slky
silt (y) ..... slt slty
siltstone ..... sltst
similar ..... sim
size ..... sz
skeletal ..... skel
slabby ..... slb
slate (y) ..... sl
slickenside (d) ..... sks
slight (ly) ..... sli
small ..... sml
smooth ..... sm
soft ..... sft
solitary ..... sol
solution soluble ..... sln
somewhat ..... smwt
sort (ing, ed) ..... srt srtg srtd
south ..... S
spar (ry) ..... spr
sparry calcite ..... spc
sparse (ly) ..... sps spsly
speck (led) ..... spk spkld
sphalerite ..... sphal
spherule (itic, s) ..... sph
spicule (ar) ..... spic
splintery ..... splty
sponge ..... spg
spore ..... spo
spot (ted, y) ..... sp sptd spty
stain (ed, ing) ..... stn
stalactitic ..... stal
stippled ..... stip
strata (ified, tion) ..... strat
streak (ed) ..... strk

(continues)

Table 8-9—(Continued)

streaming	stmg
striae (ted)	stri
stringer	strg
stromatolite (itic)	stromlt
stromatoporoid	strom
structure	struc
stylolite (itic)	styl
sub	sb
subangular	sbang
subhedral	sbhed
sublithic	sblit
subrounded	sbrd
sucrosic	suc
sugary	sug
sulphur (ous)	S su
superficial oolite (ic)	spfool
surface	surf
syntaxial	syn
tabular (ate)	tab
tan	tn
tension	tns
terriginous	ter
texture (d)	tex
thick	thk
thin section	T.S.
thin	thn
thinbedded	t.b.
throughout	thru
tight (ly)	ti
top	tp
tough	tgh
trace	tr
translucent	trnsl
transparent	trnsp
trilobite	tril
tripoli (itic)	trip
tube (ular)	tub
tuff (aceous)	tuf
type (ical)	typ
unconformity	unconf
unconsolidated	uncons
underclay	uc
underlying	undly
unidentifiable	unident
uniform	uni
upper	u
vadose	vad
variation (able ed)	var
varicolored	vcol
variegated	vgt
varved	vrvd
vein (ing ed)	vn
veinlet	vnlet
vermillion	verm
vertebrate	vrtb
vertical	vert
very poor sample	vps
very	v
vesicular	ves
violet	vi
visible	vis
vitreous (ified)	vit
volatile	volat
volcanic rock volcanic	volc
vug (gy)	vug
wackestone	wkst
washed residue	W.R.
water	wtr
wavy	wvy
waxy	wxy
weak	wk
weather (ed)	wthr
wthrd well	wl
west	W
white	wh
with	w/
without	w/o
wood	wd
yellow (ish)	yel yelsh
zeolite	zeo
zircon	Zr
zone	z

Note: Most of the terms and abbreviations are extracted from *Sample Examination Manual*, R. G. Swanson, AAPG, 1981.

Table 8-10—Standard Abbreviations for Lithologic Descriptions
(Sorted alphabetically by abbreviations.)

ditto	"
and	&
at	@
same as above	a.a.
as above	a.a.
abundant	abd
absent	abs
about	abt
above	abv
acicular	acic
after	aft
aggregate	agg
agglomerate	aglm
anhedral	ahd
algae, algal	alg
allochem	allo
alter (ed ing)	alt
altered, altering	alt
amber	amb
ammonite	amm
amount	amt
angular	ang
anhydrite (ic)	anhy
aphanitic	aph
apparent	apr
approximate (ly)	aprox
appears	aprs
aragonite	arag
arenaceous	aren
argillaceous	arg
argillite	argl
arkose (ic)	ark
asphalt (ic)	asph
assemblage	assem
associated	assoc
authigenic	authg
average	av
Barite (ic)	bar
basalt (ic)	bas
become (ing)	bcm
birdseye	bdeye
bed (ded ing)	bdg
boundstone	bdst
bentonite (ic)	bent
birefringence	bifg
bioclastic	biocl
bioherm (al)	bioh
biomicrite	biomi
biosparite	biosp
biostrom (al)	biost
biotite	biot
bioturbated	biotb
bitumen (inous)	bit
blue (ish)	bl blsh
blade (ed)	bld
boulder	bldr
black (ish)	blk blksh
block (y)	blky
basement	bm
band (ed)	bnd
bored (ing)	bor
boudinage	boudg
brachiopod	brac
brackish	brak
breccia (ted)	brec
branching	brhg
brittle	brit
break	brk
brown	brn
bright	brt
bryozoa	Bry
bottom	btm
botryoid (al)	btry
buff	bu
bubble	bubl
Bulbous	bulb
burrow (ed)	bur
core	c
calcite (ic)	CaCO3
calcareous	calc
cavern (ous)	cav
carbonized	cb
cobble	cbl
calcite filled fractures	ccf
caliche	cche
cephalopod	ceph
compare	cf
conglomerate (ic)	cgl
chalcedony (ic)	chal
charophyte	char
chitin (ous)	chit
chalk (y)	chk chky
chlorite (ic)	chlor
chocolate	choc
chert (y)	cht chty
chitinozoa	chtz
circulate (ion)	circ
clastic	clas
calcarenite	clcar
calcilutite	clclt
calcirudite	clcrd
calcisphere	clcsp
clean	cln
clear	clr
calcisiltite	clslt
cluster	clus
cleavage	clvg
clay (ey)	cly

(continues)

Table 8-10—(Continued)

claystone	clyst
cement (ed ing)	cmt
concentric	cncn
center centered	cntr
contorted	cntrt
carbonate	CO3
coal	coal
color (ed)	col
common	com
concretion (ary)	conc
conchoidal	conch
conodont	cono
considerable	cons
consolidated	consol
conspicuous	conspic
content	cont
contamination (ed)	contm
coquina (oid)	coq
coral coralline	cor corin
covered	cov
compact	cpct
crenulated	cren
crevice	crev
crinoid (al)	crin
crinal cream	crm
crinkled	crnk
cryptograined	crpgr
crumpled	crpld
cryptocrystalline	crpxl
coarse (ly ness)	crs
contact	ctc
coated (ing)	ctd
coated grains	ctd gn
cuttings	ctgs
cube (ic)	cub
caving	cvg
diabase	db
dead	dd
debris	deb
decrease (ing)	decr
dendritic	dend
depauperate	depau
description	descr
desiccation	dess
devitrified	devit
diameter	dia
diagenesis (etic)	diagn
difference	dif
disseminated	dism
distillate	dist
dark (er)	dk dkr
dense (er)	dns
dolomite (ic)	dol
dolocast (tic)	dolc
dolomold (ic)	dolmd
dolostone	dolst
dominant (ly)	dom
drilling	drig
drusy	drsy
druse	dru
drill stem test	DST
detrital (us)	dtrl
determine	dtrm
east	E
earthy	ea
echinoid	ech
elevation	elev
elongate	elg
elliptical	elip
embedded	embd
enlarged	enl
equant	eqnt
equivalent	equiv
euhedral	euhd
euxinic	eux
evaporite (itic)	evap
excellent	ex
extraclast (ic)	exclas
expose (ed ure)	exp
extrusive rock extrusive (ion)	extr
extremely	extr
fine (ly)	f fnly
frosted quartz grains	F.Q.G.
facet (ed)	fac
fauna	fau
ferruginous	Fe
iron	Fe
Ferromagnesian	Femag
fenestra (al)	fen
ironstone	Fest
fibrous	fibr
figured	fig
fissile	fis
fissures	fiss
flat	fl
flaggy	flg
flake (s y)	flk flks flky
flora	flo
fluorescence (ent)	flor
flesh	fls
fault (ed)	flt
floating	fltg
formation	fm
faint (ly)	fnt
foliated	fol
foraminifera (al)	foram
fossil (iferous)	fos
fair	fr
fracture (d)	frac
fragment (al)	frag

(continues)

Table 8-10—(Continued)

frequent ..... freq
fringe (ing) ..... frg
friable ..... fri
framework ..... frmwk
frosted ..... fros
fresh ..... frs
feldspar (athic) ..... fspr
feet ..... ft
foot ..... ft
fucoid (al) ..... fuc
fusulinid ..... fus
gas ..... G
gabbro ..... gab
gastropod ..... gast
good ..... gd
generally ..... gen
geopetal ..... gept
gilsonite ..... gil
glass (y) ..... glas
glauconite (itic) ..... glau
Globigerina (inal) ..... glob
gloss (y) ..... glos
green (ish) ..... gn gnsh
gneiss (ic) ..... gns
grain (s, ed) ..... gr
graptolite ..... grap
grapestone ..... grapst
grade (s ing d) ..... grd
granule (ar) ..... grnl
granite (ic) ..... grnt
granite wash ..... grnt.w
grainstone ..... grst
gravel ..... grv
gray, grey (ish) ..... gry grysh
greasy ..... gsy
grit (ty) ..... gt
graywacke ..... gwke
gypsum (iferous) ..... gyp
halite (iferous) ..... hal
hard ..... hd
hematite (ic) ..... hem
heterostegina ..... het
heterogeneous ..... hetr
hexagonal ..... hex
high (ly) ..... hi
hackly ..... hky
homogeneous ..... hom
hornblend ..... hornbl
heavy ..... hvy
hydrocarbon ..... hydc
horizontal ..... hztl
in part ..... I.P.
igneous rock, igneous ..... ig
impression ..... imp
inch ..... in
inclusion (ded) ..... incl
increasing ..... incr
indurated ..... ind
indistinct ..... indst
inoceramus ..... inoc
insoluble ..... insl
interbedded ..... intbd
intraclast (ic) ..... intclas
intercalated ..... intercal
intraformational ..... intfm
interfingered ..... intfr
interfragmental ..... intfrag
intergrown ..... intgn
intergranular ..... intgran
interlaminated ..... intlam
interparticle ..... intpar
interpretation ..... intpt
intrusive rock intrusive ..... intr
intraparticle ..... intrapar
intersticies (iitial) ..... intst
interval ..... intvl
intercrystalline ..... intxl
invertebrate ..... invtb
iridescent ..... irid
irridescent ..... irid
irregular (ly) ..... irr
isopachous ..... iso
jasper (oid) ..... jasp
joint (s, ed, ing) ..... jt jts jtd
kaolin (itic) ..... kao
lacustrine ..... lac
lamina (tions, ated) ..... lam
laterite (itic) ..... lat
lavender ..... lav
leached ..... lchd
ledge ..... ldg
lens, lenticular ..... len lent
long ..... lg
large larger ..... lge
lignite (itic) ..... lig
limonite (itic) ..... lim
lithic ..... lit
lithology (ic) ..... lith
lithographic ..... lithgr
littoral ..... litt
lumpy ..... lmpy
limy ..... lmy
local ..... loc
lower ..... low
limestone ..... ls
loose ..... lse
light (er) ..... lt
little ..... ltl
lustre ..... lustr
lutite ..... lut

(continues)

Table 8-10—(Continued)

layer	lyr
macrofossil	macrofos
magnetite, magnetic	mag
maroon	mar
marine	marn
massive	mass
material matter	mat
maximum	max
marble	mbl
member	mbr
mud (dy)	md mdy
median	mdn
mudstone	mdst
medium	med
meniscus	men
metamorphic (osed)	meta metaph
mica (ceous)	mic
micrograined	micgr
micromicaceous	micmica
micrite (ic)	micr
microfossil (iferous)	microfos
microoolite	microol
micropore (osity)	micropor
microspar	microspr
microstylolite	microstyl
microcrystalline	micxl
middle	mid
miliolid	milid
minimum	min
milky	mky
manganese, manganiferous	mn
minor	mnr
mineral (ized)	mnrl
minute	mnut
moderate	mod
mold (ic)	mol
mollusc (a)	moll
mosaic	mos
mottled (ing)	mot
marl (y)	mrl mrly
marlstone	mrlst
metasomatic	msm
matrix	mtrx
muscovite	musc
no show	n/s
no	n
north	N
no sample	n.s.
no visible porosity	n.v.p.
nacreous	nac
nodule (s, ar)	nod
novaculite	novac
numerous	num
oil	O
object	obj
occasional	occ
ochre	och
odor	od
olive	olv
Olivine	olvn
oncolite (oidal)	onc
ooid (al)	oo
oolicast (ic)	ooc
oolite (itic)	ool
oomold (ic)	oomol
opaque	op
orbitolina	orbit
organic	org
orange (ish)	orng
orthoclase	orth
oil source rock	OSR
ostracod	ostr
orthoquartzite	otz
overgrowth	ovgth
oxidized	ox
oyster	oyst
poor (ly)	p
pressure deformation	p d
pinpoint porosity	p.p.
paper (y)	pap
particle	par
pebble (ly, s)	pbl
pellet (al, oids)	pel
pelletoid (al)	peld
pendular (ous)	pend
permeability (able)	perm K k
petroleum, petroliferous	pet
Phenocrysts	phen
phlogopite	phlog
phyllite, phyllitic	phly
phosphate (atic)	phos
phreatic	phr
pisoid (al)	piso
pisolite, pisolitic	pisol
pitted	pit
pink	pk
pinkish	pkish
packstone	pkst
plagioclase	plag
plastic	plas
pelecypod	plcy
plant	plt
platy	plty
polish, polished	pol
pollen	poln
polygonal	poly
porosity, porous	por ϕ
porcelaneous	porcel
possible (ly)	pos
parts per million	ppm

(continues)

Table 8-10—(Continued)

predominant (ly) ..... pred
preserved (ation) ..... pres
primary ..... prim
prism (atic) ..... pris
pearl (y) ..... prly
probable (ly) ..... prob
production ..... prod
prominent (ly) ..... prom
porahyry ..... prphy
pseudo ..... psdo
pseudo oolite (ic) ..... psool
pumicestone ..... pst
part (ly) ..... pt
patch (y, es) ..... ptch
parting ..... ptg
purple ..... purp
pyrobitumen ..... pybit
pyrite (itized itic) ..... pyr
pyroclastic ..... pyrcl
pyroxene ..... pyrxn
quartz (ose, ic) ..... qtz qtzs qtzt
rounded frosted pitted ..... r.f.p.
radial (ate, ating) ..... rad
radiaxial ..... radax
rubble (bly) ..... rbl rbly
red (ish) ..... rd rdsh
recovery (ered) ..... rec
recemented ..... recem
regular ..... reg
replaced (ment, ing) ..... rep
residue (ual) ..... res
recrystal (lize, ed, ation) ..... rexl rexlzd rexlt
reef (oid) ..... rf
rhomb (ic) ..... rhb
rock ..... rk
remains remnant ..... rmn
round (ed) ..... rnd
range (ing) ..... rng
ripple ..... rpl
rare ..... rr
resinous ..... rsns
rudist ..... rud
rugose, ruga ..... rug
rugose coral, rugosa ..... rugc
salt and pepper ..... s & p
south ..... S
sulphur (ous) ..... S su
salt water ..... s.w.
sidewall core ..... S.W.C.
salt (y) ..... sa
saltcast (ic) ..... sac
saccharoidal ..... sacc
saturation (ated) ..... sat
sub ..... sb
subangular ..... sbang
subhedral ..... sbhed
sublithic ..... sblit
subrounded ..... sbrd
scales ..... sc
scaphopod ..... scaph
scatter (ed) ..... scat
schist (ose) ..... sch
scolecodont ..... scol
scarce ..... scs
sand (y) ..... sd sdy
secondary ..... sec
sediment (ary) ..... sed
selenite ..... sel
septate ..... sept
soft ..... sft
shale (ly) ..... sh shy
shadow ..... shad
shell ..... shl
shelter porosity ..... shlt por
show ..... shw
siderite (itic) ..... sid
silica (iceous) ..... sil
similar ..... sim
skeletal ..... skel
slickenside (d) ..... sks
slate (y) ..... sl
slabby ..... slb
slight (ly) ..... sli
silky ..... slky
solution soluble ..... sln
silt (y) ..... slt slty
siltstone ..... sltst
smooth ..... sm
small ..... sml
somewhat ..... smwt
solitary ..... sol
spot (ted, y) ..... sp sptd spty
sparry calcite ..... spc
superficial oolite (ic) ..... spfool
sponge ..... spg
spherule (itic, s) ..... sph
sphalerite ..... sphal
spicule (ar) ..... spic
speck (led) ..... spk spkld
sample ..... spl
splintery ..... splty
spore ..... spo
spar (ry) ..... spr
sparse (ly) ..... sps spsly
sort (ing, ed) ..... srt srtg srtd
sandstone ..... ss
stalactitic ..... stal
stippled ..... stip
streaming ..... stmg
stain (ed, ing) ..... stn

(continues)

Table 8-10—(Continued)

strata (ified, tion)	strat
stringer	strg
striae (ted)	strl
streak (ed)	strk
stromatoporoid	strom
stromatolite (itic)	stromlt
structure	struc
stylolite (itic)	styl
sucrosic	suc
sugary	sug
surface	surf
syntaxial	syn
size	sz
thinbedded	t.b.
thin section	T.S.
tabular (ate)	tab
terriginous	ter
texture (d)	tex
tough	tgh
thick	thk
thin	thn
throughout	thru
tight (ly)	ti
tan	tn
tension	tns
top	tp
trace	tr
trilobite	tril
tripoli (itic)	trip
translucent	trnsl
transparent	trnsp
tube (ular)	tub
tuff (aceous)	tuf
type (ical)	typ
upper	u
underclay	uc
unconformity	unconf
unconsolidated	uncons
underlying	undly
uniform	uni
unidentifiable	unident
very	v
vadose	vad
variation (able ed)	var
varicolored	vcol
vermillion	verm
vertical	vert
vesicular	ves
variegated	vgt
violet	vi
visible	vis
vitreous (ified)	vit
vein (ing ed)	vn
veinlet	vnlet
volatile	volat
volcanic rock volcanic	volc
very poor sample	vps
vertebrate	vrtb
varved	vrvd
vug (gy)	vug
with	w/
without	w/o
west	W
washed residue	W.R.
wood	wd
white	wh
weak	wk
wackestone	wkst
well	wl
weather (ed)	wthr wthrd
water	wtr
wavy	wvy
waxy	wxy
cross	x
crossbedded (ing)	xbd xbdg
crystal (line)	xl xln
crosslaminated	xlam
crossstratified	xstrat
yellow (ish)	yel yelsh
zone	z
zeolite	zeo
zircon	Zr

Note: Most of the terms and abbreviations are extracted from *Sample Examination Manual*, R. G. Swanson, AAPG, 1981
Units and Conversions.

Table 8-11—Units and Conversions

Quantity	Customary unit	Metric unit SPE preferred	Conversion factor multiply customary unit by factor to get metric unit	
t = time	d	s	8.6400	E+04
	s	d	1.1574	E-05
L = length	ft	m	3.048	E-01
	in	mm	2.54	E+01
A = area	sq ft	m^2	9.290	E-02
	sq in	mm^2	6.4516	E=02
V = volume, capacity	liter	dm^3	1.0	
	ft^3	m^3	2.831685	E-02
m = mass	lbm	kg	4.535942	E-01
	kg	g	1.0	E+03
	g	kg	1.0	E-03
T = temperature	˚F	˚C	(˚F - 32)/1.8	
	˚C	˚C	1.0	
	˚R	K	5/9	
	K	K	1.0	
P = pressure	atm (760 mm Hg at 0˚C or 14.696 lbf/in^2)	MPa	1.01325	E-01
		kPa	1.01325	E+02
	bar	MPa	1.0	E-01
	bar	kPa	1.0	E+02
	lbf/in^2 (psi)	MPa	6.894757	E-03
	lbf/in^2 mm Hg (0˚C)	kPa	6.894757	
	torr	kPa	1.333224	E-01
	$dyne/cm^2$	Pa	1.0	E-01
q = flow rate	ft^3/D	m^3/d	2.831685	E-02
	U.S. gal/min (liquids)	dm^3/s	6.309020	E-02
	ft^3/D	cm^3/s	3.277413	E-01
u = volumetric velocity (flux, or superficial)	ft/D	m/d	3.048	E-01
	ft/D	cm/d	3.048	E+01
	ft/D	mm/d	3.48	E+02
	ft/s	m/s	3.048	E-01
ρgas = Density (gases)	lbm/ft^3	kg/m^3	1.601846	E+01
	lbm/ft^3	g/m^3	1.601846	E+04
ρw, ρo = Density	lbm/U.S. gal (liquids)	kg/m^3	1.198264	E+02
	lbm/U.S. gal (liquids)	g/cm^3	1.198264	E-01
	lbm/ft^3	kg/m^3	1.601846	E+01
	lbm/ft^3	g/cm^3	1.601846	E-02
	g/cm^3	kg/m^3	1.0	E+03
	g/cm^3	kg/dm^3	1.0	
	˚API	specific gravity	141.5/(131.5+˚API)	
ρma = density (solids)	lbm/ft^3	kg/m^3	1.601846	E+01
ν = viscosity (Kinematic)	cm^2/s	mm^2/s	1.0	E+02
	ft^2/hr	mm^2/s	2.58064	E+01
	cST mm^2/s	1.0		
μ = viscosity (dynamic)	$dyne\text{-}s/cm^2$	Pas	1.0	E-01
	cP	Pas	1.0	E-03
	lbm/(ft-hr)	Pas	4.133789	E-04

Table 8-11—Units and Conversions (Continued)

Quantity	Customary unit	Metric unit SPE preferred	Conversion factor multiply customary unit by factor to get metric unit	
	cP	$(Ns)/m^2$	1.0	E-03
σs = surface tension	dyne/cm	m(milli)N/m	1.0	
γg = interfacial tension (IFT)	dyne/cm	m(milli)N/m	1.0	
k = absolute/specific permeability	darcy (d)	μm^2	9.869233	E-01
	millidarcy (md)	μm^2	9.869233	E-04
	microdarcy (μmd)	μm^2	9.869233	E-07
ω = angular velocity	rpm	radian/s	1.047198	E-01

Table 8-12—Nomenclature*

k_a	=	air permeability, md
k_h	=	horizontal permeability, md
k_v	=	vertical permeability, md
k_∞	=	equivalent liquid permeability (Klinkenberg corrected), md
k_{90}	=	permeability 90 degrees from horizontal permeability (k_{max}), md
k_{max}	=	higher of the k_h measured values in two directions 90 degrees apart
°C	=	degree Centigrade
°F	=	degree Fahrenheit
°R	=	degree Rankine
°K	=	degree Kelvin
cm^3	=	cubic centimeter
dm^3	=	cubic decimeter
ft	=	feet
ft^2	=	square feet
in	=	inch
m	=	meter
m^2	=	square meter
mm^2	=	square micrometer = square micron
μm^2	=	square micrometer
Pa	=	pascal
kPa	=	kilo pascal
kg	=	kilogram
g	=	gram
lbm	=	pound mass
lbf	=	pound force
lbm/ft^3	=	$pound/ft^3$
kg/m^3	=	$kilogram/cubic\ meter$
g/m^3	=	$gram/cubic\ meter$
g/cm^3	=	g/ml = gram/cubic centimeter = gram/milliliter
lbm/US	=	pound/US gallon
cST	=	centistokes
cP	=	centipoise
d	=	day
s	=	second
S_g	=	gas saturation, percent of pore volume
S_o	=	oil saturation, percent of pore volume
S_w	=	water saturation, percent of pore volume
ϕ	=	porosity, percent bulk volume
ϕ_e	=	effective porosity, percent of bulk volume
ϕ_t	=	total porosity, percent of bulk volume
PV	=	pore volume
BV	=	bulk volume
GV	=	grain volume
V_g	=	gas volume
V_o	=	oil volume
V_w	=	water volume
ρg	=	grain density
ρb	=	bulk density

*The SI Metric System of Units and SPE Metric Standard, SPE, Dallas, 1984.

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Boyle's Law Technique*

The Boyle's law method of measuring porosity is a gas transfer technique that involves the compression of gas into the pores or the expansion of gas from the pores of a clean, dry sample. Either pore volume or grain volume may be determined, depending upon the instrumentation and procedures used. It is an accurate technique when performed properly; it is fairly rapid for the majority of samples encountered, and it yields cores that can be used for further testing. It is essential that the samples be clean and dry, otherwise you will obtain erroneously low porosity values.

BULK VOLUME: The measurement of bulk volume is critical when pore volume is being calculated from the difference between bulk volume and grain volume. A good technique utilizes Archimedes' principle of displacement.

One of two applications of this principle is typically used. The first application requires that the sample be saturated with a liquid and then weighed. The sample is then submerged in the same fluid, and its submerged weight taken. The bulk volume is the difference between the two weights divided by the density of the fluid with which the core is saturated and in which it is immersed. The second application also requires that the sample be saturated with water or other suitable liquid. Water is typically used, because it can be easily vaporized from the core after the test is complete.

After saturation, the sample is immersed in a small vessel of water previously placed on the laboratory scales. For this immersion, the core sample must be suspended and lowered into the water without touching the sides of the vessel. Two weights are taken: that of the vessel and water before the core sample is immersed, and that of the vessel, water, and sample afterwards. The weight difference is equal to the weight of the water displaced by the core sample. Because the density of water is 1.0 g/cm3, this weight difference in grams is numerically equal to the bulk volume of the core sample in cubic centimeters.

Another suitable technique involves immersing the sample in mercury and measuring the volume of mercury displaced, employing a mercury pump calibrated for plug size samples.

Bulk volume can also be determined by calipering the length and diameter of a core sample and then applying appropriate mathematical formulas. Generally, information developed by calipering is not sufficiently accurate to yield valid porosities when grain volume is to be subtracted to yield pore volume. In other cases the bulk volume is determined by a direct measure of the pore volume, and this is summed with a direct measure of the grain volume.

GRAIN VOLUME: Measurement of grain volume is easily completed using a type of Boyle's law apparatus illustrated in Figure 1 (*Grain volume determination using Boyle's law*

porosimeter).

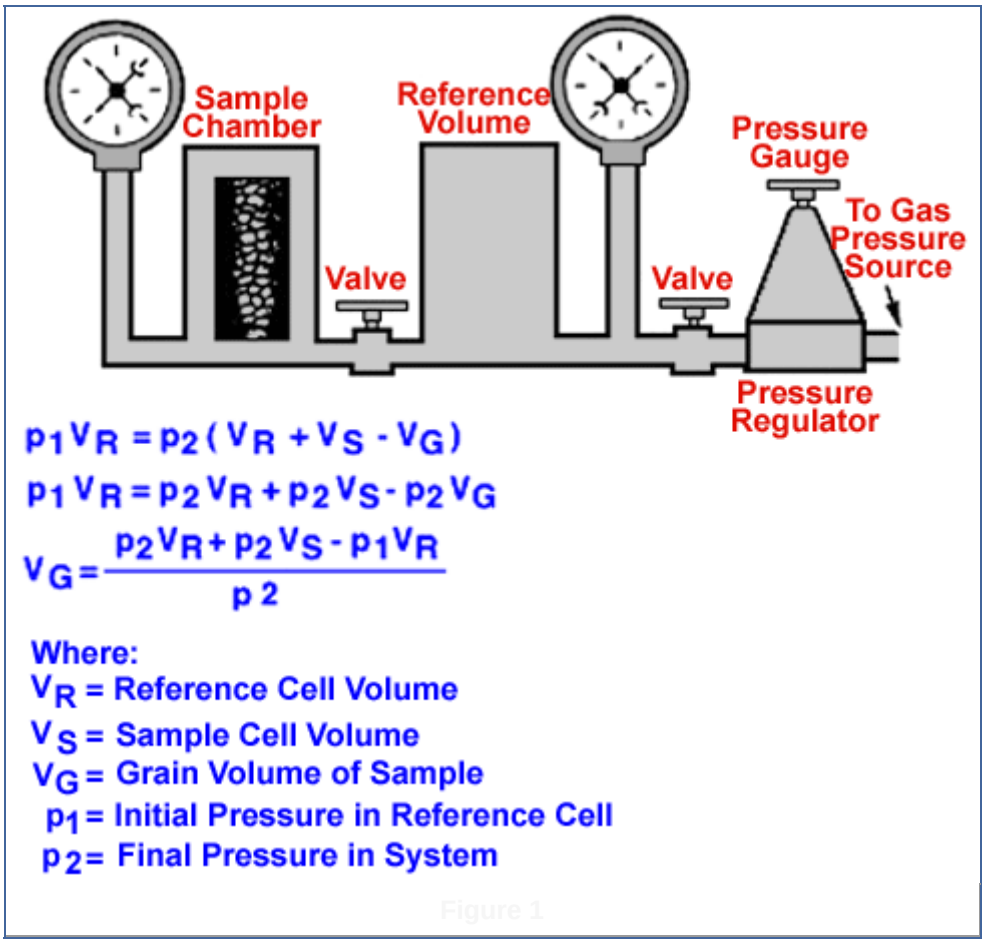

Figure 1

A clean, dry sample is placed in a chamber of known volume. This chamber is isolated from the upstream pressure chamber, which is also of known volume. The upstream pressure chamber is charged to a pressure of approximately 100 psi (689 kPa) and then isolated. The connection between the pressure chamber and sample chamber is opened and gas expands into the sample chamber, causing a drop in the original reference pressure. If volumes of the pressure and sample chamber are known, the grain volume may be calculated by using the measured pressures and the equation shown beneath Figure 1.

Precautions are necessary to secure valid data when utilizing this technique. In rocks containing free carbon and clays, air molecules can be adsorbed on the mineral surfaces, and can produce an erroneous measurement of grain volume and porosity. This limitation is overcome by using helium gas in the laboratory apparatus. Helium has an extremely small molecule that rapidly penetrates small pores. It is inert and will not be adsorbed on the rock surfaces as air can be.

Another advantage of this laboratory approach is that grain volume determined during this measurement can be subsequently combined with measured weights on the sample to yield

reliable grain density values. Incomplete cleaning and insufficient drying will yield erroneously low grain densities and erroneously high grain volumes.

PORE VOLUME: Pore volume is often determined indirectly by calculating the difference between measured values of bulk volume and grain volume. It can also be measured directly by using Boyle's law equations. In order to do this it is necessary to alter the sample holder from the configuration illustrated in Figure 1. The sample must be placed in a holder that has no void space around the periphery of the core and on the ends. An apparatus suitable for this measurement is referred to as a Hassler holder or a hydrostatic load cell. The hydrostatic load cell is illustrated in Figure 2 (*Hydrostatic load cell for direct measurement of pore volume*).

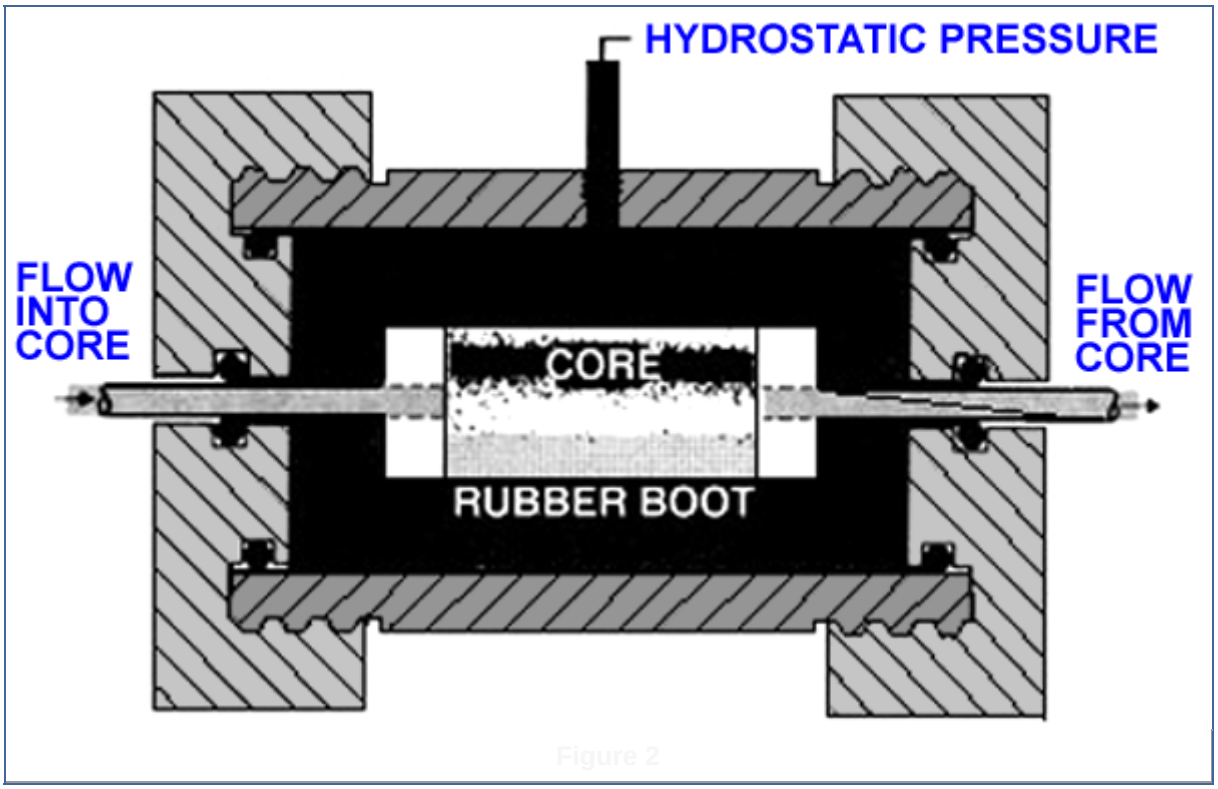

Figure 2

Helium can be injected into the core through the end stems as illustrated, and the equation beneath can be modified to furnish the sample's pore space. Dead volume in the system is measured by substituting a solid metal plug for the core plug. The sample is then inserted. It is essential that the end stems butt closely against the sample faces. If not, a dead volume not measured with the metal plug is created, which will yield an erroneously high pore space. The sample should have flat end surfaces at right angles to the axis of the core. The fact that some samples are less than perfect can be compensated for by placing a thin rubber pad (with a center hole) between the sample face and the metal end stem. The compressible rubber fills the space and allows calculation of valid data.

Parameter	**Inadequate drying***	**Properly dried**

Porosity	6.7	7.8(+ 16.4%)
Grain density	2.62	2.64(+ .02 g/cm3)
Permeability	0.03	0.06 (+50%)

*These data were rushed for well completion and used successfully for that purpose. However, this information would not be adequate for estimating reserves.

Table 1. *Example of errors in porosity, grain density, and permeability as a result of inadequate core drying*

Measurements on cores that are not clean and dry yield values of pore space that are too small. **Table 1** presents selected data measured on core samples that were rushed through the analysis without adequate time being allowed for core drying. These data were required within a short time for completion purposes, and, while adequate for that objective, would not be suitable for calculation of reserves-in-place.

Sample Prep: All tests are performed on a cylinder approximately 1 inch in diameter and about 1½ inch in length. Samples are acquired using a 1 inch hollow diamond impregnated core bit. The bit coolant is water, brine or mineral oil. The acquired core plug will be trimmed on either end using a diamond impregnated saw blade mounted onto a device equipped with an electric motor designed to cut perpendicular to the length of the core plug so as to create a near perfect cylinder.

The core plug cylinder is cleaned and dried either by Dean Stark technique using either toluene or methanol depending on the bit coolant, toluene being necessary to extract mineral oil from the core plug.

Once the core plug is cleaned, it is air dried in a convection oven at approximately 240°F for approximately 24 hours or until weight equilibrium. The dried plug is then removed and allowed to come to room temperature in a desiccator. The cooled plug is placed in a dual chambered cell, and using helium to fill the pore void, grain volume is determined. The sample is later placed in a hydrostatic core holder and the sample is confined with a minimum amount of pressure supplied by bottled nitrogen gas and using helium the pore volume is determined. The bulk volume is later determined using the Archimedes method with toluene. The sample is allowed to air dry and is then oven dried.

* Joe I found this article on the internet at this location: (http://ipims.com/data/fe32/E3034.asp?UserID=&Code=3229) and it seemed appropriate for your application. I've edited a few items into the file that apply to the sample prep.

APPENDIX B: ACCIDENT PREVENTION PLAN/SITE SAFETY AND HEALTH PLAN/ACTIVITY HAZARD ANALYSES

REMEDIAL INVESTIGATION / FEASIBILITY STUDY SUPPLEMENTAL INVESTIGATION

ACCIDENT PREVENTION PLAN, SITE SAFETY AND HEALTH PLAN, AND ACTIVITY HAZARD ANALYSIS

FORMER NIKE BATTERY PR-58
NORTH KINGSTOWN, RHODE ISLAND
REMEDIAL INVESTIGATION / FEASIBILITY STUDY

FUDS Project Number: D01RI0029 05

CENAE Project Number: 108346

Contract Number: W912WJ-11-D-0001

Stone Project ID: 102305-R

October 2013

Prepared for:

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STONE ENVIRONMENTAL INC

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1. SIGNATURE SHEET

HEALTH AND SAFETY PLAN SIGNATURE SHEET
FOR REMEDIAL INVESTIGATION / FEASIBILITY STUDYAT THE
NIKE BATTERY PR-58 SITE,
NORTH KINGSTOWN, RHODE ISLAND

Prepared under the direction of: Kim Watson 28 Oct 2013
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Prepared by:

Stone Environmental, Inc. Montpelier, Vermont
Task Order 0001 Contract : W912WJ-11-D-0001

2. BACKGROUND INFORMATION

2.1. APPLICABILITY

This Accident Prevention Plan/Site Safety and Health Plan/Activity Hazard Analysis (APP/SSHP/AHA) has been developed by Stone Environmental, Inc. (Stone) for the US Army Corps of Engineers New England District (USACE) under Task Order 0001 of Contract W912WJ-11-D-0001. It establishes the health and safety procedures to minimize any potential risk to Stone personnel and their subcontractors conducting Stone Field Tasks at the Nike Battery PR-58 Site in North Kingstown, Rhode Island. The proposed field work will support the ongoing overburden investigation for the evaluation of the nature and extent of chlorinated solvent contamination in soils, groundwater, surface water, and sediment due to former military activities. Additional tasks that are expected to be necessary for a remedial investigation are also included. Stone is conducting this work on behalf of USACE.

The provisions of this plan apply to all Stone personnel and Stone subcontractor personnel who may potentially be exposed to safety and/or health hazards related to activities described in this document. Some of the work is to be performed during the 2012. This APP/SSHP/AHA only addresses the specific tasks being implemented by Stone and Stone subcontractors.

This APP/SSHP/AHA has been written to comply with the requirements of the Occupational Safety and Health Administration (OSHA) Hazardous Waste Operations and Emergency Response Standard (29 CFR 1910.120) and the USACE Safety and Health Requirements Manual (EM 385-1-1 November 2003). This APP/SSHP/AHA also directly incorporates the USACE requirements for an Accident Prevention Plan (APP) as outlined in Appendix A of the USACE Safety and Health Requirements Manual. All activities covered by this APP/SSHP/AHA must be conducted in complete compliance with this APP/SSHP/AHA and with all applicable federal, state, and local health and safety regulations. Personnel covered by this APP/SSHP/AHA who cannot or will not comply will be excluded from site activities.

This plan will be distributed to each employee involved with the proposed investigative program being implemented by Stone. Each employee must sign a copy of the attached health and safety plan receipt and acceptance form (see Attachment A).

2.2. SITE LOCATION

The Property is located in the North Kingstown Quonset Business Park, and encompasses approximately 39 acres. The property is bounded by the former Perimeter Road to the north, Seabee Avenue to the east, Newcomb Road to the west, and Babcock Road to the south (Figure 1). The areas to the north and west of the Property are predominantly residential with minimal, light commercial/industrial land use. The property is owned by the Quonset Development Corporation (QDC). To the south of the Property is the QDC commercial development of the former Quonset Point Naval Air Station (NAS) and to the east is the former Davisville Naval Construction Battalion Center (NCBC). The NAS site and NCBC sites are the focus of their own contamination investigations and remediation planning. The Site encompasses area outside of the property that may have been impacted by contaminants originating on the Property. The boundaries of the Site are not known at this time.

2.3. HISTORICAL INFORMATION

The primary contaminants of concern at the Site have been identified during previous investigations as chlorinated volatile organic compounds (CVOCs). Of particular interest is the distribution of the primary CVOCs detected in groundwater: 1,1,2,2-tetrachloroethane (perchloroethane [TeCA]); 1,1,2-trichloroethane (1,1,2-TCA); tetrachloroethene (perchloroethene [PCE]); trichloroethene (TCE); cis-1,2 dichloroethene (cis-1,2-DCE); trans-1,2 dichloroethene (trans-1,2-DCE); 1,1 dichloroethene (1,1- DCE), and vinyl chloride (VC).

The contamination includes:

- Soil contamination:

  The near-surface and vadose zone soils have shown very few detections of organic contaminants in past investigations. The most significant detections were of the CVOCs and the highest soil concentrations are noted at the locations where the highest groundwater concentrations have been measured. Soil samples in the Property source area taken from the lower overburden unit had the highest CVOC concentrations. TeCA, TCE, and cis-1-2 DCE are the contaminants with the highest concentrations. In the near surface (less than 2 feet below ground surface (fbgs) soils, no CVOC have been detected. At depths up to ten ft bgs, the only reported CVOC is TeCA, at a concentration of an estimated 0.002J mg/kg, orders of magnitude less than the Rhode Island Residential Direct Exposure Criteria concentration of 1.3 mg/Kg. Contaminated soils below 10 feet expected to be encountered during drilling may contain concentrations approaching 100% saturation.

- Non-Aqueous Phase Liquids:

  Evidence of TeCA and TCE NAPL has been found in the deep overburden and bedrock on the former Nike property.

- Groundwater (overburden):

  Groundwater at the Property and off-Property has been impacted by a number of CVOCs including: TeCA, PCE, TCE, cis- and trans-1,2 DCE, 1,1 DCE, 1,1,2 TCA and vinyl chloride. The CVOC with the highest reported concentration is TeCA. TeCA degrades into 1,1,2-TCE, TCE, cis- and trans-1,2 DCE and many of the other observed CVOCs on-Property. Maximum reported historical concentrations of these chemicals in groundwater are as follows:

Table 1: Maximum Reported Historical CVOC Concentrations in Groundwater

Compound	Maximum Concentration (ppb)
TeCA	330,000
1,1,2 TCA	8,000
PCE	4,540
TCE	177,000
cis- -1,2 DCE	22,100
trans-1,2 DCE	7,800
1,1 DCE	1,780
vinyl chloride	159

2.4. CONTRACTOR ACCIDENT EXPERIENCE

Stone maintains a file of a summary of work-related injuries and illnesses via OSHA's Form 300A. Completed forms are maintained on file at Stone's Corporate office in Montpelier, Vermont. A summary for the 2011 calendar year maintains that out of the 67,990 total hours worked, with an average number of employees at 40, the total number of days away from work due to an injury or illness was zero (0).

2.5. PURPOSE OF INVESTIGATION

The primary goal of the work is to address the data gaps identified in the Data Gap Analysis Report, May 2012. The work will use a combination screening results from a mobile laboratory and fixed laboratory analysis.

2.6. FIELD TASKS

The field work will include:

- Perform site reconnaissance, collect surface geophysical data; perform surveying;
- Collect pore water, sediment and surface water samples for fixed laboratory analysis;
- Collect and analyze groundwater samples from approximately 50 monitoring wells;
- Collect and analyze drinking water samples from approximately 12 potable water locations;
- Perform subsurface soil sampling with drilling equipment;
- Chemical Analysis in Mobile Field Laboratory;
- Install and develop groundwater monitoring wells;
- On-site handling of Investigation-Derived Wastes;
- Equipment and personnel decontamination.

Figure 1: Property Location Map

3. STATEMENT OF SAFETY AND HEALTH POLICY

Stone has provided quality engineering and environmental consulting services since 1992. The safety and health of employees has always been, and will continue to be, a top priority of Stone. Stone has an excellent safety record, and intends to maintain that record.

Stone has developed and implemented a Health and Safety Program (HASP) to ensure continuation of safe work practices, further emphasize health and safety awareness, and comply with Occupational Safety and Health Administration (OSHA) workplace regulations and guidelines.

The APP/SSHP/AHA presents a general description of health and safety risks associated with the type of work performed by employees of Stone along with Standard Operating Procedures to be followed for prevention and protection.

When work tasks are performed at a specific project site other than Stone's office or other fixed facilities (equipment and material storage), an APP/SSHSP/AHA shall be prepared and used in conjunction with the corporate HASP.

4. RESPONSIBILITIES AND LINES OF AUTHORITY

4.1. ORGANIZATION/RESPONSIBILITIES

The implementation of health and safety at this project location will be the shared responsibility of the Stone PM, Stone Health and Safety Officer, Site Safety Officers (SSO) and other staff and contractors participating in this field program.

Table 2: Stone Contacts

Title	SSO	Name, Firm	Phone Number	Cell Number	Email
Project Manager	X	Joseph Schmidl	802-229-5377	802-477-3126	jschmidl@stone-env.com
Site Safety Officer	X	Lee Rosberg	802-229-6434	802-309-1629	lrosberg@stone-env.com
Corporate Health and Safety Officer		Kim Watson	802-299-2196	802-249-7753	kwatson@stone-env.com

Project Manager (PM)

The Stone PM for this project is Joseph Schmidl.

The PM(s) are individuals who are primarily responsible for ensuring the overall health and safety of this project. As such, the PM(s) are responsible for ensuring that the requirements of this APP/SSHP/AHA are implemented. Some of the PM's specific responsibilities include:

- Assuring that all personnel to whom this APP/SSHP/AHA applies have received a copy;
- Providing the team Health and Safety Officers with updated information regarding conditions at the Site and the scope of site work;
- Supporting the decisions made by the SSO and team health and Safety Officers;
- Maintaining regular communications with the SSOs and, if necessary, the team health and Safety Officers; and,
- Coordinating the activities of all team subcontractors and ensuring that they are aware of the pertinent health and safety requirements for this project.

Corporate Health and Safety Officer

Stone's Corporate Health and Safety Officer for this project is Kim Watson. Her credentials are provided in Attachment E.

As the Corporate Health and Safety Officer, she is responsible for the preparation, interpretation and modification of this APP/SSHP/AHA. Modifications to this APP/SSHP/AHA which may result in less stringent precautions cannot be undertaken by the PM or the SSO without the approval of the Health and Safety Officer. Specific duties of the team Health and Safety Officers include:

- Develop a site specific APP/SSHP/AHA designed to provide the necessary safeguard to sufficiently protect employees working at the site;
- Ensure that the APP/SSHP/AHA addresses all compliance issues associated with the work at the site, to include both regulatory and contractual requirements;
- Review the final APP/SSHP/AHA to ensure that all foreseeable hazards have been identified and that reasonable hazard control systems are in place;
- Advising the PM and SSOs on matters relating to health and safety on this site;

- Recommending appropriate personal protective equipment (PPE) to protect team personnel and team subcontractors from potential site hazards;
- Conducting accident investigations; and,
- Maintaining regular contact with the PM and SSO to evaluate site conditions and new information which might require modifications to the APP/SSHP/AHA.

Site Safety Officer (SSO)

The SSO for this project is Lee Rosberg. His credentials are provided in Attachment E. All field staff are responsible for implementing the safety requirements specified in this APP/SSHP/AHA; however, only one field staff member will serve as the SSO. The SSO will be appointed by the PM. The SSO for any given day of field work will be on-site during all team activities covered by this APP/SSHP/AHA. The SSO is responsible for enforcing the requirements of this APP/SSHP/AHA once work begins. The SSO has the authority to immediately correct all situations where noncompliance with this APP/SSHP/AHA is noted and to immediately stop work in cases where an immediate danger is perceived. Some of the SSO's specific responsibilities include:

- Assuring that all personnel to whom this APP/SSHP/AHA applies have submitted a completed copy of the APP/SSHP/AHA receipt and acceptance form;
- Assuring that all personnel to whom this APP/SSHP/AHA applies have attended a pre-entry briefing and any subsequent safety meetings that are conducted during the implementation of this program;
- Interfacing and coordinating site access and work activities with the USACE site representative and, if necessary, other on-site contractors working for the USACE;
- Maintaining a high level of health and safety consciousness among team members and team contractors at the work site;
- Procuring and distributing the PPE needed for this project for team field members;
- Verifying that all PPE and health and safety equipment used by team members is in good working order;
- Verifying that the selected team contractors are prepared with the PPE and safety equipment required by this APP/SSHP/AHA;
- Notifying the PM of all noncompliance situations and stopping work in the event that an immediate danger situation is perceived;
- Monitoring and controlling the safety performance of team personnel and team contractors within the established restricted areas to ensure that required safety and health procedures are being followed;
- Conducting accident/incident investigations and preparing accident/incident investigation reports;
- Conducting the pre-entry briefing as required by the APP/SSHP/AHA; and,
- Initiating emergency response procedures in accordance with this APP/SSHP/AHA.

Field Staff

All team field personnel covered by this APP/SSHP/AHA are responsible for following the health and safety procedures specified in this APP/SSHP/AHA and for performing their work in a safe and responsible manner. Some of the specific responsibilities of the field personnel are as follows:

- Reading the APP/SSHP/AHA in its entirety prior to the start of on-site work;
- Submitting a completed APP/SSHP/AHA Acceptance Form to the team SSO prior to the start of work;
- Attending the required pre-entry briefing prior to beginning on-site work and any subsequent safety meetings that are conducted during the implementation of the proposed field program;
- Bringing forth any questions or concerns regarding the content of the APP/SSHP/AHA to the PM or the SSO prior to the start of work;
- Immediately correct all situations where noncompliance with this APP/SSHP/AHA is noted and to immediately stop work in cases where an immediate danger is perceived;
- Reporting **all** accidents, injuries and illnesses, regardless of their severity, to the team SSO; and
- Complying with the requirements of this APP/SSHP/AHA and the requests of the SSO.

5. SUBCONTRACTORS AND SUPPLIERS

Stone has retained subcontractors in order to complete the requirements of the project. A description of the subcontractors who will be working in the field and their responsibilities is presented below. All subcontractors are responsible for their own safety and concurrence with OSHA and project specific requirements. The roles of the Stone subcontractors are as follows:

Geophysics

Geophysical services for subsurface utility clearance purposes will be provided by Hager-Richter Geoscience, Inc. Jeff Reid is the contact for surface geophysics. Their contact information is provided below:

Hager-Richter Geoscience
Jeff Reid
8 Industrial Way – D10
Salem, New Hampshire 03079
Phone: 603-893-9944
Jeff.Reid@hager-richter.com

Drilling

Drilling services will be provided by:

Stone Environmental Inc.
Platform Environmental Drilling and Remediation Services
535 Stone Cutters Way
Montpelier, Vermont 05602
Phone: 802-229-1883
mjordan@stone-env.com

Surveyor

Surveying services will be provided by Environmental Planning and Surveying, Inc. 523 Dugway Bridge Road, West Kingstown, Rhode Island. Phone 401-789-3628.

On-site Subcontractors involved in intrusive work hired by Stone are responsible for:

- Reading the APP/SSHP/AHA in its entirety prior to the start of on-site work;
- Attending the required pre-entry briefing prior to beginning on-site work and any subsequent safety meetings that are conducted during the implementation of the program;
- Ensuring, via daily inspections, that their equipment is in good working order;
- Operating their equipment in a safe manner;
- Appointing an on-site safety coordinator to interface with the Stone SSO;
- Providing the SSO with copies of training certifications and material safety data sheets (MSDS) for all hazardous materials brought on-site; and,
- Providing all the required PPE, respiratory equipment and safety supplies to their employees.

6. TRAINING

6.1. Medical monitoring

All personnel performing activities covered by this APP/SSHP/AHA, except for the field reconnaissance, surface geophysics and surveying, must be active participants in the medical monitoring program that complies with 29 CFR 1910.120(f). Each individual must have completed an annual surveillance examination within the last year (if exposure criteria are met) and an initial baseline examination prior to performing any work on the site covered by this APP/SSHP/AHA.

6.2. Health and Safety Training

6.2.1. HAZWOPER

All personnel performing on-site activities, except for the field reconnaissance, surface geophysics and surveying, covered by this APP/SSHP/AHA must have completed the appropriate training requirements specified in 29 CFR 1910.120(e). Each individual must have completed an annual 8-hour refresher-training course and/or initial 40-hour training course within the last year prior to performing any work on the sites covered by this APP/SSHP/AHA. Additionally, each employee will have completed at least three days of on-site training under the direction of a trained and experienced team supervisor from the member's employer. Also, on-site managers and supervisors directly responsible for supervising individuals engaged in hazardous waste operations must have completed the specified 8-hour managers training course.

6.2.2. Hazard Communication

Hazard communication training is incorporated into the Stone HAZWOPER training program. The specific hazards associated with the calibration gases and equipment decontamination solutions will be reviewed during the pre-entry briefing. MSDSs for these materials will be maintained on site.

6.2.3. First Aid/CPR

At least two members of the field team will be certified in First Aid/CPR. In addition, staff have had trainng on controlling exposure to blood-borne pathogens and the use of universal precautions.

6.3. Pre-Entry Briefing

The SSO will conduct a pre-entry briefing to all personnel on-site before site activities begin, to include subcontractor employees from the onsite laboratories. Specific topics that will be discussed during the pre-entry briefing include:

- Discussion of site history
- Discussion of work scope
- Review of the potential hazards associated with contaminants of concern and how these potential hazards will be controlled
- Review of air monitoring requirements and action limits
- Review of PPE and respiratory protection requirements
- Discussion of the potential physical hazards associated with implementing the scope of work
- Review of emergency egress and hospital location/directions

- Review of decontamination procedures

APP/SSHP/AHA receipt and acceptance sheets will be collected at this meeting. Short safety refresher meetings will be conducted daily during on-site field work throughout the duration of the project. Attendance of the pre-entry meeting and daily safety refreshers is mandatory and will be documented by the SSO. An attendance form is presented in Attachment C.

7. SAFETY AND HEALTH INSPECTIONS

As discussed in other places in this APP/SSHP/AHA, all employees have the right to stop work if deficiencies are noted and only to resume work once these deficiencies have been corrected. In most cases, identified issues will be easy to resolve on site.

Field inspections (audits) will be conducted by the PM and/or the Corporate Health and Safety Officer or designee. For short duration jobs (i.e., one week or less), the safety inspection should be conducted shortly after the work has commenced. For longer duration jobs, the inspection should be conducted shortly after commencement and monthly thereafter, if applicable.

Audits should be documented on the PM Audit Checklist. The auditor should print out a copy of the checklist before leaving for the job site, unless a computer and printer will be available at the site. Once the inspection is complete, the form should be placed in the project files.

In the event that deficiencies are noted during the audit, the PM, SSO and the Health and Safety Officer will confer as necessary to determine how to correct the deficiencies and how to determine when work can resume. The SSO will provide follow-up to insure that the deficiencies have been corrected.

8. SAFETY AND HEALTH GOALS AND OBLECTIVES, INCENTIVE PROGRAMS, AND COMPLIANCE

The objectives of the safety and health program at Stone, specifically as required under the contract with the USACE, are to develop and provide the necessary documents which describe the health and safety criteria, procedures, practices and equipment to be implemented and utilized during the performance of field tasks to sufficiently protect on-site personnel from chemical, physical, and/or biological hazards that may be encountered. Specific goals of the safety and health program and site-specific health and safety plans are to prevent unacceptable exposure to chemicals, protect against physical and environmental hazards, and avoid accidents that could injure personnel.

Strict adherence to Stone's health and safety program, and site-specific health and safety plans, is considered part of each employee's obligations working at Stone. Any overt noncompliance with health and safety procedures is treated as non-performance under the terms of employment, which is dealt with promptly through a personal meeting between the employee and executive management of Stone. The employee is warned that another such noncompliance incident could lead to termination of employment depending on the circumstances. If the incident was inadvertent or occurred due to a lack of understanding or knowledge, the meeting will be with the Corporate Health and Safety Officer, and will be used to develop strategies to avoid future incidents.

Managers and Senior Management are held accountable for the proper implementation of all health and safety procedures for projects/employees they are in charge of by ensuring that their role and responsibilities are well defined in the HASP and SSHP. Their awareness of the level of accountability they maintain is enhanced by ensuring they read (and sign where appropriate) the appropriate plans, and through periodic health and safety meetings with Company Management, the Corporate Health and Safety Officer, and other PMs.

9. ACCIDENT REPORTING

9.1. Stone Environmental, Inc. Procedures

Any work related injury or illness must be reported by the worker to the Corporate Health and Safety Officer and executive management within 24 business hours of the accident/incident by completing the reporting section of Stone's Incident/Accident/Injury/Illness Reporting Form (Attachment D) . The Corporate Health and Safety Officer in conjunction with the QA representative shall investigate either incident/accident/injury/ illness/ and fill out the investigation section of the form with the injured employee. The Safety Committee will review this report. If the accident/incident may have been preventable the Corporate Health and Safety Officer and QA representative shall indicate by what means future accidents/incidents of a similar nature could be avoided. The Corporate Health and Safety Officer and QA representative shall also investigate if applicable health and safety procedures were followed, whether safety and/or personal protective equipment was used and was the usage proper. The completed form will be kept on file for at least five years.

9.2. USACE Reporting Requirements

All accidents and near misses shall be investigated by the SSO. All work-related recordable injuries, illnesses and property damage accidents (excluding on-the-road vehicle accidents), in which the property damage exceeds $2,000 shall be reported verbally to the USACE by the SSO or PM within 24 hours of the incident. Serious accidents as described in EM 385-1-1 Section 01.D.02 shall be immediately reported to the USACE. ENG Form 3394 shall be completed and submitted to the USACE within five working days of the incident.

The PM shall complete the USACE Contractor Monthly Summary Record of Injuries/Illness and Work Hour Exposure (for the prime contractor and its subcontractors) and forward the completed form to the USACE no later than close of business on the 10th calendar day of the following month. The method of transmission by the prime contractor to the USACE shall be electronically.

10. MEDICAL SUPPORT

Fire extinguishers, eye wash equipment, and a first aid kit shall be maintained on-site at all times that personnel are performing tasks on-site. The location of these facilities will be shown to all on-site personnel during the health and safety briefings described above.

An ambulance, the fire department, and the police can be contacted by dialing 911 on a cell phone.

The nearest hospital is approximately 10 miles away:

Kent County Memorial Hospital
455 Tollgate Road
Warwick, RI 02886
401-737-7000

The SSO or his designee is responsible for arranging off-site emergency medical care and transport. Mike Jordan and Lee Rosberg are trained in CPR and First Aid (see Attachment E).

Directions to Hospital (see Figure 2):

From PERIMETER RD.

Turn RIGHT onto DAVISVILLE RD.

At the traffic circle, take the 3rd EXIT onto the RI-403 W ramp.

Keep RIGHT at the fork, follow signs for RI-4 N/ I-95/ Providence and merge onto RI-4 N.

RI-4 N becomes I-95 N.

Take EXIT 10 for RI-117 toward Warwick/ Warwick W

Turn RIGHT onto CENTERVILLE RD/ RI-117 E.

Turn LEFT onto TOLL GATE RD/ RI-115 W.

End at 455 Toll Gate Rd Warwick, RI 02886

11. MONITORING INSTRUMENTS AND PERSONAL PROTECTIVE EQUIPMENT

An Activity Hazard Analysis (AHA) has been prepared for each of the anticipated tasks (see Attachment B).

11.1. Direct Reading Instrumentation

11.1.1. Photoionization Detector (PID)

An Ionscience Phocheck PID (model 1000+), with a 10.6 eV lamp will be used to monitor the breathing zone by the SSO during the proposed subsurface investigations to determine the presence of VOCs. If the PID indicates sustained (15 minute) breathing zone vapor concentrations in excess of 1 ppmV or more in the breathing zone above background concentrations, engineering controls will be implemented or respiratory protection, as described this document, will be donned.

The PID will be calibrated daily to 100 parts per million (ppm) isobutylene span gas and to a zero level (clean air reference) in accordance with Stone's standard operating procedure *SEI-6.19.2 Use, Maintenace and Calibration of the Ionscience Phocheck 1000+ Photionization Detector (PID)*. Daily calibration information will be recorded in the field notebook or on appropriate forms.

11.2. Personal Protective Equipment (PPE)

As required by OSHA 1910.120, Personal Protective Equipment (PPE) must be selected that will protect team members from the specific hazards they are likely to encounter on-site. Selection of the appropriate PPE shall be in general compliance with 29 CFR 1910.132. In areas where the potential for contact with the contaminants of concern exist, Modified Level D will be worn unless air monitoring indicates that the action level set for respiratory exposure is exceeded. If this occurs, engineering controls will be implemented, and/or Level C will be worn as long as the respirator and cartridge is capable of reducing the level of potential exposure to workers to below the Permissible Exposure Level (PEL) or Threshold Limit Value (TLV).

Stone's Corporate Health and Safety Officer has reviewed the contaminants of concern and the proposed tasks and have completed PPE assessments for each task and have reviewed applicable manufacturer's charts to determine that the selected chemical protective clothing is appropriate for the contaminants of concern at this site

11.2.1. Chemical Protective Clothing

PPE will be worn during all field activities to prevent on-site personnel from being injured by the safety hazards or exposed to hazardous chemicals activities. The following describes the different circumstances PPE to be worn under during performance of site.

11.2.1.1. Level D or Modified Level D

Level D or Modified Level D PPE are the most likely PPE levels that will be required during this work. It should be cautioned that PPE selection and evaluation is an ongoing process and may be adjusted as additional information about the hazards at the job site and PPE performance is obtained.

The Level D ensemble includes long pants, shoes and a shirt. The Level D protection may be modified as follows:

- Type I or Type II (ANSIZ89.1) hard hat (when overhead hazards are present)
- Safety glasses with permanently attached sideshields (when splash, dust, flying particles or other eye hazards are present)
- Safety-toed boots compliant with the ANSI Z41 standard (when working with heavy machinery)
- Cut-resistant gloves (when using knives or blades, or working with power tools and machinery)
- Chemically-resistant gloves when collecting samples, handling investigation derived wastes, or performing work with potential contact hazards with chemicals (i.e. nitrile for short exposure times of less than five minutes and Viton for longer exposure times). Gloves should not be re-used.
- Hearing protection (when excessive noise levels exist)
- Traffic Vest (when working around heavy machinery such as loaders, excavators, trucks or backhoes)
- Tyvek coverall (as need to protect clothing and skin from splash or chemical contact hazards)
- Lab coats for personnel working in the on-site laboratory
- Personal floatation device when working from small boat or near water hazard

11.2.1.2. Level C or Modified Level C

Based on detectible contaminant concentrations and the expected locations and types of activities, it is not currently anticipated that Level C PPE will be necessary in the conduct of the field work. However, Level C PPE is included in this APP/SSHP/AHA to protect workers in the event unanticipated vapor levels (in excess of the Action Level) are encountered during intrusive field activities at known or suspected source areas.

The Level C ensemble includes the following:

- Half mask or full mask air purifying respirators (NIOSH approved) with organic vapor cartridges (safety glasses required if half mask respirator is used).
- Tyvek coverall
- Gloves, outer, chemical-resistant.(Viton)
- Gloves, inner.
- Inner boots, chemical-resistant safety toe and shank.
- Disposable boot-covers, outer, chemical-resistant (optional)
- Hard hat (when overhead hazards exist).
- Hearing protection (when excessive noise levels exist).

11.2.1.3. Respiratory Protection

If the PID indicates sustained (15 minute) breathing zone VOC vapor concentrations in the breathing zone in excess of 1 ppmV above background levels, engineering controls will be implemented and/or respiratory protection will be donned.

Engineered air exchanges are used to provide controls for vapors in the mobile laboratory.

Level C Specification: Half- mask or full-mask, air-purifying respirator with organic vapor cartridges

All employees who are expected to don respiratory protection must have been successfully fit-tested for the model, brand and size respirator they plan to wear at this site within the past year.

11.2.1.4. Other Protective Equipment

The following additional safety equipment will be brought on-site by team personnel:

- Portable hand-held eyewash bottles that will deliver at least 0.4 gallons of water per minute with a minimum of six gallons of water available on-site
- First aid kit
- Type A-B-C fire extinguisher (on drilling equipment and in vehicles),
- Portable walkie-talkies and/or cell phones
- Class 3 lime yellow traffic vest (compliant with ANSI/ISEA 107 Class 3 high visibility apparel).

Potable water and sanitary facilities will be provided on-site for hand washing, eye washing (if needed), etc.

11.2.1.5. Summary of Required PPE for Specific Tasks

Table 3 presents a summary of the minimum required PPE level on an activity-specific basis.

Table 3: Required Personal Protective Equipment by Activity

Activity No.	Activity Description	PPE Level
1	Reconnaissance, surface geophysics and surveying	Level D
2	Pore water, sediment and surface water sample collection	Modified Level D (with personal floatation device, as needed)
3	Collect and analyze groundwater samples from monitoring wells	Modified Level D
4	Collect and analyze drinking water samples.	Modified Level D
5	Perform subsurface soil sampling with drilling equipment	Modified Level D (with hard hats, hearing protection, safety boots, gloves and safety glasses)
6	Chemical Analysis in Mobile Field Laboratory	Modified Level D (with lab coats, gloves and safety glasses)
7	Install and develop groundwater monitoring wells	Modified Level D
8	On-site handling of Investigation-Derived Wastes	Modified Level D
9	Equipment and Personal Decontamination	Modified Level D

By participating in OSHA's 40-hr HAZWOPER safety training, as well as subsequent 8-hr HAZWOPER refresher courses, each employee who is required to wear PPE for this field program has been properly instructed as to how to inspect and maintain their safety gear. Prior to mobilizing to the site, each employee will inspect their safety gear. Any equipment that is defective, such as scratched safety glasses or cracked hard hats, will be replaced. Chemical protective clothing such as gloves will be inspected by each wearer for holes, rips and tears. Defective clothing will be thrown away.

12. PLANS REQUIRED BY THE SAFETY MANUAL

The following sections of this APP/SSHP/AHA incorporate details of the plans required by the safety manual, including:

- An emergency response plan, (18.0)
- Spill and firefighting plans, (18.4)
- Emergency phone numbers, (10.0)
- Hazard communication, (18.3)
- Respiratory protection, (11.2)
- Lifting procedures, (15.6)
- Severe weather contingencies, (15.7)
- Prohibitions on alcohol and drug use, (16.3)
- Slip, trip and falls, and (15.1)
- Site sanitation. (16.0 and 17.0)

13. CONTRACTOR INFORMATION

The requirements of applicable sections of the EM 385-1-1 are addressed throughout this document. The APP/SSHP/AHA has been reviewed along-side-of EM 385-1-1, and adjusted as necessary to be in accordance with the manuals requirements.

14. SITE-SPECIFIC HAZARDS AND CONTROLS

14.1. Chemical Hazards and Contaminants of Concern

During its years of operation, the Army reportedly used solvents, including TCE. The Navy reportedly used TeCA containing chemicals on the property. Based on standard practices at the time, it is assumed that waste solvents were disposed of on the ground at the various locations where they were used and eventually migrated into the groundwater.

Solvents have been detected in groundwater and soils on the Property. The potential exists for exposure to TeCA, PCE, TCE, cis- and trans-1,2 DCE, 1,1 DCE, 1,1,2 TCA and vinyl chloride during intrusive work. MSDS sheets for these compounds are presented in Attachment F.

Overexposure to the chlorinated organic solvents detected in groundwater may result in depression of the central nervous system, symptoms of which include dizziness, headache, giddiness and drunken-like behaviors. Chronic overexposures can result in liver and kidney damage.

A summary of the Contaminants of Concern and their associated exposure limits is presented in Table 4.

Table 4: Contaminants of Potential Concern and Associated Exposure Threshold and Physical Properties

Chemical	PEL[1] (ppmV)	TLV[2] (ppmV)	Odor Threshold (ppmV)	IDLH (ppmV)	Density (water = 1) @ 20°C (g/ml)	Henry's Constant @ 25°C (atm-m^3/mol)	Vapor Pressure @ 25°C (mmHg)	Vapor Density @ 20°C (Air = 1) (g/cm^3)	Solubility in H_20@ 25°C (mg/l)	Log K_{ow}	Maximum Reported Conc. in Groundwater µg / ml
1,1,2,2-tetrachloroethane (TeCA)	5	1	0.233-7.9	100	1.60	$3.67x10^{-4}$	4.6	1.60	$2.83x10^3$	2.39	330,000
Tetrachloroethene (PCE)	100	25	47	500	1.62	$1.8x10^{-2}$	18.5	1.62	$1.5x10^2$	3.40	4,540
Trichloroethene (TCE)	100	50	82	1,000	1.46	$1.1x10^{-2}$	74	1.47	$1.366 x10^3$	2.42	177,000
cis-1,2-dichloroethene (cis-1,2-DCE)	200	200	0.08-17	1,000	1.28	$3.37x10^{-3}$	180	1.28	$3.5 x10^3$	1.86	22,100
trans-1,2-dichloroethene (trans-1,2-DCE)	200	200	0.08-17	1,000	1.26	$6.72x10^{-3}$	265	1.26	$6.3 x10^3$	2.09	7,800
1,1-dichloroethane (1,1-DCA)	100	100	49-1,359	3,000	1.18	$4.2x10^{-2}$	230	1.17	$5.5 x10^3$ *	1.79	8,000
Vinyl chloride (VC)	1	5	10-20		0.91	$2.78x10^{-2}$	2,600	2.16	$2.763 x10^3$	1.36	159

[1] Permissible Exposure Limit – OSHA; 8 hour time weighted average (8 hr.- TWA)
[2] Threshold Limit Value – American Conference of Governmental Industrial Hygienists; 8 hr.- TWA
Sources: ATSDR Toxic Profiles, NIOSH Pocket Guide to Chemical Hazards, 3-M Respirator Guide

14.2. Hazardous Substances Brought Onsite by Team Members

In accordance with OSHA's Hazard Communication Standard (29 CFR 1910.1200) as well as Section 01.B.06C of EM385-1-1, a material safety data sheet (MSDS) must be available for each hazardous substance that team members bring on the Site. This includes solutions/chemicals that will be used to decontaminate sampling equipment and any calibration gases or calibration reagents that team members may use for field instruments. Once on site, the SSO will create an inventory of all hazardous substances brought on site. This inventory will include the name of the material, the estimated quantity on site and a general description of where these materials are being stored. In accordance with Stone's Hazard Communication Program, team members will be informed of the hazardous properties of the chemicals with which they may work, safe handling procedures, and measures to take to protect them from chemical hazards. MSDS sheets for these materials are located within the facilities and/or mobile units, which will be maintained current during all site work. All containers of hazardous materials must be properly labeled in accordance with OSHA's Hazard Communication Standard.

14.3. Chemical Exposure and Control

The potential exists for workers to be exposed to contaminants of concern through inhalation of vapors and direct dermal contact with contaminated soils, rock, and/or groundwater.

The following chemical exposure control measures will be implemented during the proposed investigations:

- To protect against exposure to COCs through inhalation of vapors, a photoionization detector (PID) will be utilized to record sustained readings. Should readings exceed 1ppmV as an average of three measurements over a fifteen-minute interval, engineering controls will be implemented to reduce the level to less than 1 ppmV, and/or a respirator will be used by personnel within the vapor zone. Should peak readings exceed 100 ppmV, workers will evacuate the area and notify the SSO and/or the PM.
- To avoid direct dermal contact with contaminated media, protective clothing will be required when collecting samples, decontaminating sampling equipment, and installing or removing data loggers.
- To avoid direct dermal contact with contaminated media, protective clothing, including lab coats, chemical resistant gloves and eye protection will be required when analyzing samples in the field laboratory.
- Although highly unlikely, exposure to all of the contaminants of concern may occur via ingestion (hand-to-mouth transfer). The decontamination procedures address personal hygiene issues that will limit the potential for contaminant ingestion.
- The field work program does not involve activities that will expose workers to contaminated air-borne particulates, with the possible exception of the air rotary drilling.

15. PHYSICAL HAZARDS AND CONTROLS

15.1. Slips, Trips, and Falls

On any work area, it is expected that the ground may be uneven. The ground surface may be unreliable due to settling. Surface debris may be present and wet or swampy areas may exist.

Extension cords and hoses may be present. Cords and hoses should be routed so that they lay flat on the ground, and are long enough that they do not cross heavily traveled areas.

Tools such as shovels, rakes, and other hand tools will be used. Tools and equipment should not be left on the ground when not in use. They should be placed in a tool box, on a tool belt, or in specially designated and clearly marked areas. Orange or similar bright colored survey flagging may be attached to the tool handles to make them clearly visible.

Employees should walk around, not over or on top of debris or trash piles. When carrying equipment, identify a path that is clear of any obstructions. It may be necessary to remove obstacles to create a smooth, unobstructed access point to the work areas.

15.1.1. Working in Steep Terrain

Workers may be walking over rough terrain to perform the proposed sampling. This work may occur when temperatures, high winds and inclement weather, may make traversing the area difficult. Employees must be cautious when working not to become overzealous and try to quickly access areas on steep slopes.

All work will be conducted by a two-person team. The team will be equipped with appropriate communications equipment to facilitate contact between the team and the USACE on-site representative and/or the local emergency responders in the event of an emergency.

During winter months, each team member will be equipped with appropriate cold weather gear, as well as shoes equipped with strap-on, spike-like attachments that provide traction on ice during the winter, such as Yaktrax™.

15.1.2. Good Housekeeping

The active portion of the work area should be maintained in a neat and clean condition to reduce the opportunity for tripping when walking.. Maintaining a work environment that is free from accumulated debris is the key to preventing slip, trip and fall hazards at construction sites. Essential elements of good housekeeping include:

- orderly placement of materials, tools and equipment;
- placing trash receptacles at appropriate locations for the disposal of miscellaneous rubbish;
- prompt removal and secure storage of items that are not needed to perform the immediate task at hand;
- awareness on the part of all employees to walk around, not over or on, equipment that may have been stored in the work area.

15.1.3. Water Hazards

The collection of surface water, pore water, and sediment samples will require working in and around surface waters. A canoe or small boat may be required to collect samples in some waters. All work on or near surface waters and from small boats will be conducted with a minimum of two people. Personal floatation devices

will be worn when working from small boats. The SSO will determine when other potential water hazards near work areas require workers to don personal floatation devices.

15.2. Working in Close Proximity to Operating Machinery

Stone will be working around and in close proximity to drill rigs. The use of such equipment poses a potential hazard to the support crew working around the equipment. Use of heavy equipment at the site requires all employees working in the exclusion zone to wear ANSI-approved Type I or Type II hard hats, safety-toed shoes/boots, safety glasses and hearing protection.

15.2.1. Working in Close Proximity to Drilling Equipment

On-site personnel working in close proximity to drilling equipment will be required to observe the following safety rules as well as wear the PPE discussed in the paragraphs above.

- Personnel shall not remain in the vicinity of operating equipment unless it is required for their work responsibilities.
- All drill rigs and other machinery with exposed moving parts must be equipped with an operational emergency stop device. Drillers and geologists must be aware of the location of this device. This device must be tested prior to job initiation and periodically thereafter.
- Drillers, helpers and geologists must secure all loose clothing when in the vicinity of drilling operations to avoid entanglement.
- Make eye or verbal contact with operator to insure that the rig is idle when entering work area, when approaching the drill string or hoist, when placing or removing blocking, and when operating auger racks to avoid being struck, entangled, or pinched by operating equipment

15.3. Noise

Working in close proximity to drill rigs may expose Stone employees to noise levels that exceed the OSHA PEL of 90 dBA for an 8-hour day. Exposure to noise can result in the following:

- Temporary hearing losses where normal hearing returns after a rest period;
- Interference with speech communication and the perception of auditory signals;
- Interference with the performance of complicated tasks; and
- Permanent hearing loss due to repeated exposure resulting in nerve destruction in the inner ear.

Although personal noise monitoring will not be conducted during the proposed activities, it is well established that drilling rigs and other heavy equipment can emit sound levels beyond the OSHA established limits. The table below outlines likely noise levels that can be experienced during site activities. Disposable earplugs or earmuffs with a minimum noise reduction rating (NRR) of 27 db will be worn anytime Stone employees are subjected to 85dBA or above in accordance with Table 5 below.

Table 5: RCNM Default Noise Emission Reference Levels and Usage Factors

Equipment Description	Impact Device?	Acoustical Usage Factor (%)	Spec. 721.560 L_{max} @ 50 feet (dBA, slow)	Actual Measured L_{mx} @ 50 feet (dBA, slow) (Samples Averaged)	Number of Actual Data Samples (Count)
All Other Equipment > 5 HP	No	50	85	N/A	0
Auger Drill Rig	No	20	85	84	36
Backhoe	No	40	80	78	372
Bar Bender	No	20	80	N/A	0
Blasting	Yes	50	85	N/A	0
Boring Jack Power Unit	No	50	80	83	1
Chain Saw	No	20	85	84	46
Clam Shovel (dropping)	Yes	20	93	87	4
Compactor (ground)	No	40	80	78	18
Compressor (air)	No	50	85	N/A	0
Concrete Batch Plant	No	15	83	N/A	0
Concrete Mixer Truck	No	40	85	79	40
Concrete Pump Truck	No	20	82	81	30
Concrete Saw	No	20	90	90	55
Crane	No	16	85	81	405
Dozer	No	40	85	82	55
Drill Rig Truck	No	20	84	79	22
Drum Mixer	No	50	80	80	1
Dump Truck	No	40	84	76	31
Excavator	No	40	85	81	170
Flat Bed Truck	No	40	84	N/A	0
Front End Loader	No	40	80	79	96
Generator	No	50	82	81	19
Generator(<25KVA, VMS Signs)	No	50	85	N/A	0
Gradall	No	40	85	83	70
Grader	No	40	85	N/A	0
Grapple (on backhoe)	No	25	80	82	6
Hydra Break Ram	Yes	10	90	N/A	0
Impact Pile Driver	Yes	20	95	101	11
Jackhammer	Yes	20	85	89	133
Man Lift	No	20	85	75	23

Equipment Description	Impact Device?	Acoustical Usage Factor (%)	Spec. 721.560 L_{max} @ 50 feet (dBA, slow)	Actual Measured L_{mx} @ 50 feet (dBA, slow) (Samples Averaged)	Number of Actual Data Samples (Count)
Mounted Impact Hammer (hoe ram)	Yes	20	90	90	212
Pavement Scarifier	No	20	85	90	2
Paver	No	50	85	77	9
Pickup Truck	No	40	55	75	1
Pneumatic Tools	No	50	85	85	90
Pumps	No	50	77	81	17
Refrigerator Unit	No	100	82	73	3
Rivet Buster/Chipping Gun	Yes	20	85	79	19
Rock Drill	No	20	85	81	3
Roller	No	20	85	80	16
Sand Blasting (single nozzle)	No	20	85	96	9
Scraper	No	40	85	84	12
Sheers (on backhoe)	No	40	85	96	5
Slurry Plant	No	100	78	78	1
Slurry Trenching Machine	No	50	82	80	75
Soil Mix Drill Rig	No	50	80	N/A	0
Tractor	No	40	84	N/A	0
Vacuum Excavator (Vac-Truck)	No	40	85	85	149
Vacuum Street Sweeper	No	10	80	82	19
Ventilation Fan	No	100	85	79	13
Vibrating Hopper	No	50	85	87	1
Vibratory Concrete Mixer	No	20	80	80	1
Vibratory Pile Driver	No	20	95	101	44
Warning Horn	No	5	85	83	12
Welder/Torch	No	40	73	74	5

Reference: Reherman, Clay M., Rochat, Judith L., Thalheimer, Erich S., Lau, Michael C. Fleming, Gregg C., Ferroni, Mark and Corbisier, Christopher. Roadway Construction Noise Model (RCNM). Federal Highway Administration, Washington, DC: January 2006.

15.4. Air Compressor Use

Compressed air or nitrogen may be used to drill and develop wells, for geophysical work, or to inflate packers. Compressed air may also be used to inflate bladder pumps used for groundwater sampling. When using an air compressor or compressed nitrogen, the following safety requirements must be implemented:

- Operators shall perform a pre-operational check of all air hoses, couplings and connections to determine if leakage or other damage exists. Be familiar with operator's manual. Do not operate unsafe equipment.
- All pipes, hoses, and fittings must have a rating of the maximum pressure of the compressor.
- Air supply shutoff valves should be located (as near as possible) at the point-of-operation.
- Air hoses should be kept free of grease and oil to reduce the possibility of deterioration.
- Hose ends must be secured to prevent whipping if an accidental cut or break occurs.
- Pneumatic impact tools should never be pointed at a person.
- Before a pneumatic tool is disconnected (unless it has quick disconnect plugs), the air supply must be turned off at the control valve and the tool bled.
- Compressed air must not be used under any circumstances to clean dirt and dust from clothing or off a person' s skin. When directed against the skin, even 30 psi of compressed air can be driven into the flesh or eyes. If there is a break in the skin, air can be driven into tissue, causing swelling and pain. Bubbles of air can be driven into blood vessels, which can be serious, even fatal.
- Goggles, face shields or other eye protection must be worn by personnel using compressed air for cleaning equipment.

15.5. Cuts and Lacerations

Minor wounds are the most likely of the risks to persons moving about the project site. Cuts and abrasions are most likely to occur as individuals move through trees and underbrush and contact thorny plants, sharp stones, sharp ends of branches, and similar objects. Many of these injuries can be prevented by wearing appropriate clothes, gloves, and footwear. Eye injuries can be prevented by wearing safety glasses, or goggles. Most of these types of injuries require only minor first aid administered on site. Medical attention should be sought for more serious injuries.

Employees are at an increased risk of cutting themselves with the knives used to cut tubing for groundwater sampling. Tube-cutters are available and should be used to eliminate this hazard. If knives or blades must be used, follow the safety precautions listed below:

- Keep your free hand out of the way
- Secure your work if cutting through thick material
- Use only sharp blades; dull blades require more force which results in less knife control
- Don't put your knife in your pocket
- Use a self-retracting blade
- Wear leather or Kevlar® gloves when using knives or blades.

15.6. Back Safety

The manual moving and handling of materials on a site poses a risk to workers in the form of muscle strains and minor injuries due to dropped containers. Using the proper techniques to lift and move heavy pieces of equipment is important to reduce the potential for back injury. The following precautions should be implemented when lifting or moving heavy objects (generally defined as fifty pounds or more under ”perfect” conditions, and less if loading or unloading vehicles, if the weight is not evenly distributed in the object, or if the ground conditions are not level, smooth and dry):

- Use mechanical devices to move objects that are too heavy to be moved manually.
- If mechanical devices are not available, ask another person to assist you.
- Bend at the knees, not the waist. Let your legs do the lifting.
- Do not twist while lifting.
- Bring the load as close to you as possible before lifting.
- Be sure the path you are taking while carrying a heavy object is free of obstructions and slip, trip and fall hazards.

15.7. Thermal Stress

Work in Rhode Island could present both cold and heat stress concerns.

15.7.1. Cold Stress

15.7.1.1. Types of Cold Stress

Cold injury is classified as either localized, as in frostbite, frostnip or chilblain; or generalized, as in hypothermia. The main factors contributing to cold injury are exposure to humidity and high winds, contact with wetness and inadequate clothing. Hypothermia may appear at temperatures above freezing, especially if the person becomes wet from precipitation, sweating, or immersion in water.

The likelihood of developing frostbite increases when the face or extremities are exposed to a cold wind in addition to cold temperatures. The freezing point of the skin is about 30° F. When fluids around the cells of the body tissue freeze, skin turns white. This freezing is due to exposure to extremely low temperatures. As wind velocity increases, heat loss is greater and frostbite will occur more rapidly.

15.7.1.2. Symptoms of Cold Stress

Symptoms of hypothermia, a condition of abnormally low body temperature, include uncontrollable shivering and sensations of cold. The heartbeat slows and may become irregular, the pulse weakens and the blood pressure changes. Pain in the extremities and severe shivering can be the first warning of dangerous exposure to cold.

Maximum severe shivering develops when the body temperature has fallen to 95° F. Productive physical and mental work is limited when severe shivering occurs. Shivering is a serious sign of danger. Immediately remove any person who is shivering from the cold.

The first symptom of frostbite is usually an uncomfortable sensation of coldness, followed by numbness. There may be a tingling, stinging or aching feeling in the affected area. Patches of white may be visible on exposed skin. The most vulnerable parts of the body are the nose, cheeks, ears, fingers and toes.

15.7.1.3. Cold Stress Monitoring Techniques

- Mental Function: Monitor personnel for decreases in mental function as exhibited by fatigue, forgetfulness, reduced alertness, increase in minor injuries, etc. Any worker showing signs of decreased mental function shall be placed in a warm environment for a minimum period of 10 minutes. If symptoms persist, the worker shall be released of work that further exposes them to cold until approved by the SSO.
- Physical Signs: If any worker complains of serious discomfort or exhibits discoloration of lips or extremities, numbness, or excessive shivering, the worker shall be moved to a warm location until symptoms abate. The worker shall not be allowed to work on any task that further exposes them to the cold until approved by the SSO.

15.7.1.4. Methods to Prevent Cold Stress

When the ambient temperature, or a wind chill equivalent, falls to below 40° F (American Conference of Governmental Industrial Hygienists recommendation), site personnel who must remain outdoors should wear insulated coveralls, insulated boot liners, hard hat helmet liners and insulated hand protection. Wool mittens are more efficient insulators than gloves. Keeping the head covered is very important, since 40% of body heat can be lost when the head is exposed. If it is not necessary to wear a hard hat, a wool knit cap provides the best head protection. A facemask may also be worn.

Persons should dress in several layers rather than one single heavy outer garment. The outer piece of clothing should ideally be wind and waterproof. Clothing made of thin cotton fabric or synthetic fabrics such as polypropylene is ideal since it helps to evaporate sweat. Polypropylene is best at wicking away moisture while still retaining its insulating properties. Loosely fitting clothing also aids in sweat evaporation. Denim is not a good protective fabric. It is loosely woven which allows moisture to penetrate. Socks with high wool content are best. If two pairs of socks are worn, the inner sock should be smaller and made of polypropylene or similar types of synthetic material that wick away moisture. If clothing becomes wet, it should be taken off immediately and a dry set of clothing put on.

If wind conditions become severe, it may become necessary to shield the work area temporarily. The SSO and the PM will determine if this type of action is necessary. Heated break trailers or a designated area that is heated should be available if work is performed continuously in the cold at temperatures, or equivalent wind chill temperatures, below 20° F.

Dehydration occurs in the cold environment and may increase the susceptibility of the worker to cold injury due to significant change in blood flow to the extremities. Drink plenty of fluids, but limit the intake of caffeine.

15.7.2. Heat Stress

15.7.2.1. Types of Heat Stress

Heat related problems include heat rash, fainting, heat cramps, heat exhaustion and heat stroke. Heat rash can occur when sweat isn't allowed to evaporate, leaving the skin wet most of the time and making it subject to irritation. Fainting may occur when blood pools to lower parts of the body and as a result, does not return to the heart to be pumped to the brain. Heat related fainting often occurs during activities that require standing erect and immobile in the heat for long periods of time. Heat cramps are painful spasms of the muscles due to

excessive salt loss associated with profuse sweating. Heat exhaustion results from the loss of large amounts of fluid and excessive loss of salt from profuse sweating. The skin will be clammy and moist and the affected individual may exhibit giddiness, nausea and headache.

Heat stroke occurs when the body's temperature regulatory system has failed. The skin is hot, dry, red and spotted. The affected person may be mentally confused and delirious. Convulsions could occur. EARLY RECOGNITION AND TREATMENT OF HEAT STROKE ARE THE ONLY MEANS OF PREVENTING BRAIN DAMAGE OR DEATH. A person exhibiting signs of heat stroke should be removed from the work area to a shaded area. The person should be soaked with water to promote evaporation. Fan the person's body to increase cooling.

15.7.2.2. Early Symptoms of Heat-Related Problems

- decline in task performance
- excessive fatigue
- loss of coordination
- reduced vigilance
- decline in alertness
- muscle cramps
- unsteady walk
- dizziness

Susceptibility to Heat Stress Increases due to:

- lack of physical fitness
- obesity
- lack of acclimation
- drug or alcohol use
- increased age
- sunburn
- dehydration
- infection

People unaccustomed to heat are particularly susceptible to heat fatigue. First timers in PPE need to gradually adjust to the heat.

15.7.2.3. The Effect of Personal Protective Equipment

Sweating normally cools the body as moisture is removed from the skin by evaporation. However, the wearing of certain personal protective equipment (PPE), particularly chemical protective coveralls (e.g., Tyvek), reduces the body's ability to evaporate sweat and thereby regulate heat buildup. The body's efforts to maintain an acceptable temperature can therefore become significantly impaired by the wearing of PPE.

15.7.2.4. Measures to Avoid Heat Stress

The following guidelines should be adhered to when working in hot environments:

- Establish work-rest cycles (short and frequent are more beneficial than long and infrequent).
- Identify a shaded, cool rest area.
- Rotate personnel, alternate job functions.
- Water intake should be equal to the sweat produced. Most workers exposed to hot conditions drink less fluid than needed because of an insufficient thirst. DO NOT DEPEND ON THIRST TO SIGNAL WHEN AND HOW MUCH TO DRINK. For an 8-hour workday, 50 ounces of fluids should be drunk.
- Eat lightly salted foods or drink salted drinks such as Gatorade to replace lost salt.
- Save most strenuous tasks for non-peak heat hours such as the early morning or at night.
- Avoid double shifts and/or overtime.

The implementation and enforcement of the above mentioned measures will be the joint responsibility of the PM and SSO. Potable water should be made available each day for the field team.

15.7.2.5. Heat Stress Monitoring Techniques

Site personnel should regularly monitor their heart rate as an indicator of heat strain by the following method: Check radial pulse rates by using fore-and middle fingers and applying light pressure to the pulse in the wrist for one minute at the beginning of each rest cycle. If the pulse rate exceeds 110 beats/minute, shorten the next work cycle by one-third and keep the rest period the same. If, after the next rest period, the pulse rate still exceeds 110 beats/minute, shorten the work cycle by one-third.

Table 6 provides guidelines for work-rest regimens, but does not replace the self-monitoring techniques described above. Work activity levels (work load) in Table 6 are based on the following ACGIH categories:

Rest includes sitting quietly in place or sitting with moderate arm movements, and is in the same environment as the work activity. Relocation to a shaded environment and/or removal of personal protective equipment to allow for more effective cool-down between work periods may be considered where possible.

Light work includes sitting with moderate arm and leg movements; standing with light work at a machine or bench while using mostly arms or with some walking about.

Moderate work includes vigorous arm movement in a standing position; walking about with moderate lifting or pushing; walking on level at 6 km/hr while carrying a 3 kg weight load.

Heavy work includes heavy arm efforts such as hand sawing carpentry; shoveling dry sand; heavy assembly work on a non-continuous basis; intermittent heavy lifting pushing or pulling (e.g., pick-and-shovel work).

Very heavy work includes strenuous work such as shoveling wet sand.

Table 6: Typical Work-Rest Cycles for Warm Weather Conditions

		ACCLIMATIZED				UNACCLIMATIZED			
	Work Demands	Light	Moderate	Heavy	Very Heavy	Light	Moderate	Heavy	Very Heavy
ONE CLOTHING LAYER	100% Work	85.1°F	81.5°F	78.8°F	N/A	81.5°F	77.0°F	72.5°F	N/A
	75% Work 25% Rest	86.9°F	83.3°F	81.5°F	N/A	84.2°F	79.7°F	76.1°F	N/A
	50% Work 50% Rest	88.7°F	85.1°F	83.5°F	81.5°F	86.0°F	82.4°F	79.7°F	77.0°F
	25% Work 75% Rest	90.5°F	87.8°F	86.0°F	85.1°F	87.8°F	84.2°F	82.4°F	79.7°F
TWO LAYERS	100% Work	78.8°F	75.2°F	72.5°F	N/A	75.2°F	70.7°F	66.2°F	N/A
	75% Work 25% Rest	80.6°F	77.0°F	75.2°F	N/A	77.9°F	73.4°F	69.8°F	N/A
	50% Work 50% Rest	82.4°F	78.8°F	77.0°F	75.2°F	79.7°F	76.1°F	73.4°F	70.7°F
	25% Work 75% Rest	84.2°F	81.5°F	79.7°F	78.8°F	81.5°F	77.9°F	76.1°F	73.4°F
THREE LAYERS	100% Work	76.1°F	72.5°F	69.8°F	N/A	72.5°F	68.0°F	63.5°F	N/A
	75% Work 25% Rest	77.0°F	74.3°F	72.5°F	N/A	75.2°F	70.7°F	67.1°F	N/A
	50% Work 50% Rest	79.7°F	76.1°F	74.3°F	72.5°F	77.0°F	73.4°F	70.7°F	68.0°F
	25% Work 75% Rest	81.5°F	78.8°F	77.0°F	76.1°F	78.8°F	75.2°F	73.4°F	70.7°F

15.8. Biological Hazards

Work is scheduled for the late fall. Some of the project locations are wooded and the potential does exist for encountering poisonous plants, insects, and spiders.

15.8.1. Poisonous Plants

Persons working on the Site should be aware of the presence of poisonous plants. **Poison ivy** is a climbing plant with leaves that consist of three glossy, greenish leaflets. Poison ivy has conspicuous red foliage in the fall. Small yellowish-white flowers appear in May through July at the lower leaf axils of the plant. White berries appear from August through November. Poison ivy is typically found east of the Rockies. **Poison oak** is similar to poison ivy but its leaves are oak-like in form. Poison oak occurs mainly in the south and southwest. **Poison sumac** typically occurs as a small tree or shrub and may be 6-20 feet in height. The bark is smooth, dark and speckled with darker spots. Poison sumac is typically found in swampy areas and east of the Mississippi. The leaves have 7-13 smooth-edged leaflets and drooping clusters of ivory-white berries appear in August and last through spring.

The leaves, roots, stems and fruit of these poisonous plants contain oil called urushiol. Contact with the irritating oil causes an intensely itching skin rash and characteristic, blister-like lesions. The oil can be transmitted on soot particles when burned and may be carried on the fur of animals, equipment and apparel.

Proper identification of these plants is the key to preventing contact and subsequent dermatitis. Wear long sleeves and pants when working in wooded areas. In areas of known infestation, wear Tyvek coveralls and gloves. Oils are easily transferred from one surface to another. If you come in contact with these poisonous plants, wash all exposed areas immediately with cool water, detergent solution, or alcohol saturated wipes to remove the oils. Some commercial products such as Tecnu's Poison Oak-n-Ivy Cleanser claim to further help with the removal of oils.

15.8.2. Insects, Spiders

Insects, spiders, bees, and wasps can be prevalent during certain times of the year. Field personnel should try to avoid contact by surveying the area in which they will be walking, standing, sitting, leaning, grabbing, lifting, or reaching. Workers are encouraged to wear insect repellent when working in areas where insects are expected to be a hazard.

- Insect bites or stings - Many insects bite or sting, but few can cause serious effects by themselves, unless the person is hypersensitive to contact. Bee, wasp, and hornet hives may be present in some buildings and active during certain times of the year. The insect stings are a hazard to personnel who are hypersensitive. Site personnel with known or suspected sensitivity should carry their “bee sting kit,” which normally includes antihistamine and epinephrine to counter anaphylactic reactions.
- Although rare the only spiders dangerous spiders found in the Northeast are the black widow and brown recluse spiders - The black widow spider has a shiny black body about the size of a pea, and has a red or yellow hourglass-shaped mark on its abdomen. It weaves shapeless webs in undisturbed areas, such as woodpiles. A bite may result in severe pain, illness, or death (usually from complications and not from the bite itself). Only the female is poisonous; the male is harmless, The brown recluse is golden brown with a dark brown or black "fiddle," which is often shiny. They range from 5-15 mm (1/4 to 3/4 inch) long. Brown recluse spiders are found

primarily in the South and Midwest. They are rarely, if ever, found in Rhode Island. Brown recluse spiders live in basements and garages of houses, often hiding behind boards and boxes. Bites sometimes occur when the spiders hide in towels or clothing. They are seldom aggressive and bite only when threatened or injured. The severity of the bite of the brown recluse may vary from no symptoms at all to a reaction that is very severe. Often there is a systemic reaction within 24-36 hours of the bite characterized by restlessness, fever, chills, nausea, weakness and joint pain.

- Rocky Mountain Spotted Fever - Bites of wood ticks may result in the transmission of Rocky Mountain Spotted Fever, a serious and possibly fatal disease caused by the Rikettsia virus. The Rikettsia virus infects wood ticks, which may bite humans, thereby transferring the virus into the bloodstream. Rocky Mountain Spotted Fever occurs mostly in the late spring and early summer, and is characterized by chills, fever, severe pain in the leg muscles and joints, and a body rash.
- Lyme Disease - Female deer ticks are about 1/16 inch in length. If a tick is not removed, or if the tick is allowed to remain for days feeding on human blood, a condition known as tick paralysis can develop. This is due to a neurotoxin, which the tick apparently injects while engorging. This neurotoxin acts upon the spinal cord causing loss of coordination, weakness and paralysis. Lyme disease is most often characterized with a "bull's-eye" rash, accompanied by nonspecific symptoms such as fever, malaise, fatigue, headache, muscle aches, and joint aches. PPE offers some protection against insects, but personnel should also perform a self-search at the end of each day to check for ticks if they have been working in areas where ticks may be prevalen. To remove a tick, grasp the head, not the body, firmly with tweezers and gently pull the head out. If the bite is irritated treat it with antiseptic.

15.9. Military and Explosive Constituents

No military ordnance has been found at the Nike Battery PR-58 to date. The Nike missiles contained conventional warheads and the warheads were removed with the missiles. The military police were would have been armed and the largest weapon system they would have had is a light machine gun. Grenades, mortars, artillery, nuclear-biological-chemical munitions, land mines, missiles and bombs are not expected at this site. However, if during the course of investigation ordnance is observed, team members will:

- Stop work immediately and leave the ordnance in place.
- Secure the work area with yellow hazard tape
- Leave the work area
- Contact Army Corps of Engineers Project Manager (Casey Haskell, 978-318-8398).

16. SITE CONTROL

16.1. Workzones

To prevent both exposure of unprotected personnel and migration of contamination due to tracking by personnel or equipment, work areas along with personal protective equipment requirements will be clearly identified. The SSO will designate work areas or zones as suggested in the "Occupational Safety and Health Guidance Manual for Hazardous Waste Site Activities," NIOSH/OSHA/USCG/EPA, November 1985. They recommend the areas surround each of the work areas to be divided into three zones:

- Exclusion or "hot" zone
- Contamination Reduction Zone (CRZ)
- Support Zone

16.1.1. Exclusion Zone

Exclusion zones will be established around any work area where heavy equipment, such as an excavator or drill rig, is used. These zones will be demarcated with traffic cones and/or safety warning tape. All employees entering an exclusion zone or any work area must be prepared to don the required PPE for the work tasks being conducted in that area.

16.1.2. Contamination Reduction Zone

The decontamination zone will be established adjacent to the exclusion zone for the decontamination of the drill rig and tools that are in contact with the potentially contaminated soil, rock, and groundwater. Traffic cones, signs, and warning tape will be utilized as needed to restrict access to the decontamination station when not in use.

A personnel decontamination zone for personnel will be established adjacent to each immediate work area. Personnel will remove contaminated gloves and other disposable items in this area and place them in a plastic bag until they can be properly disposed of.

16.1.3. Support Zone

At this site, the support zone will include the team member field vehicles.

16.2. Site Communication

The Nike Battery PR-58 Site is not located at a remote site. Cell phones, and landlines will be used to facilitate contact between team members and the USACE on-site representative and/or local emergency response personnel in the event of an emergency.

16.3. General Field Work Rules

These rules shall be observed for all tasks conducted (as appropriate) at any project site.

- The "buddy system" shall be used at all times by field personnel in the exclusion zone.
- Visual, voice or radio communications shall be maintained at all times.
- Contact with contaminated surfaces, water, soils, and equipment shall be avoided whenever possible. Individuals shall not walk on discolored surfaces or kneel on the ground, nor lean, sit or place equipment on drums or containers.

- Eating, drinking, and smoking are prohibited in exclusion and decontamination zones. Alcoholic beverages and illegal drugs are prohibited on the job site.
- Hands and face must be thoroughly washed upon leaving a decontamination area. Further, whenever decontamination procedures for outer garments are in effect, the entire body shall be thoroughly washed as soon as possible after the protective garment is removed.
- Failure to complete a respiratory fit test shall preclude admission to any zone requiring respirator wear.
- All equipment must be decontaminated or discarded upon exiting an exclusion zone.
- Personal protective equipment shall be required for all field personnel and may include, but is not limited to, respiratory protection, hard hat, chemical resistant coveralls, boots, gloves, safety glasses, and ear protection.
- Anyone reporting to work under the influence of alcohol and/or illegal drugs will not be allowed access to the site. Any employee taking prescription medication or under a physician's care must notify the SSO prior to reporting to work.
- All persons shall listen for and yield the right-of-way to construction equipment.
- All equipment operators shall provide warning prior to movement and watch for personnel in their path.
- Employees are responsible for cleaning and maintaining the protective equipment issued to them. Any and all defects or failures of the equipment shall be reported immediately to the SSO.
- All personnel shall report all injuries and/or illnesses to the SSO regardless of the severity of injuries.
- Personnel shall avoid any potentially dangerous environmental situations such as entering a confined space without proper supervision, training and equipment.
- "Dig Safe" must be notified at least 72 hours prior to beginning excavation work.
- All personnel are required to contact the PM when conducting a field investigation or site inspection. At a minimum, they must check in and out of the Site with the PM or designee. If no other project personnel are on the site, the field person shall call the PM or Site Safety Officer at the home office prior to initiating field tasks and immediately after completing them. It is advisable to notify the PM at the time of the initial call of the estimated time required to complete the tasks.
- Personnel encountering a potentially hazardous environment (e.g., noticing strong vapor levels of unidentified substances) shall instruct all other on-site personnel to leave the Site and shall call the PM and SSO for instruction. Personnel shall not re-enter the Site without proper protective clothing, and shall not work at the Site until the unknown substance is identified and characterized.
- A NIOSH approved hard hat shall be worn at all times when overhead hazards are present.
- A shirt and long pants shall be worn at all times.
- Goggles shall be worn when handling chemicals that may damage the eyes.
- Skin abrasions must be thoroughly protected to prevent chemical exposure.

- Each employee is responsible for his or her health and safety.

17. DECONTAMINATION

17.1. Personnel Decontamination

Proper decontamination is required of all personnel before leaving the site. Decontamination will occur within the decontamination zone. Disposable PPE will be removed in the decontamination zone and placed in garbage bags. All disposable PPE will be disposed of as solid waste.

Regardless of the type of decontamination system required, potable water and liquid soap should be made available so employees can wash their hands before leaving the Site for lunch or for the day.

Potable water will be brought to the daily work location by the work vehicles. Portable sanitary facilities will be provided near work zones on the Property.

17.2. Equipment Decontamination

Hand-held equipment will be decontaminated prior to introducing it into a well, between wells, and after completion of field work at a particular well or sampling point. Decontamination will generally consist of a tap water rinse to remove gross contamination, followed by a non-phosphate detergent water wash and rinse, an isopropanol rinse, and finally a triple de-ionized or distilled water rinse.

Heavy equipment will be decontaminated as specified in the work plan. The drill rig(s), drill tools, and associated equipment shall be cleaned with a high temperature and pressure, hot water sprayer prior to commencement and after completion of drilling at each location.

The equipment decontamination shall be performed at the area designated adjacent to the work location. Potable water for decontamination will be obtained from an approved source.

17.3. Investigation-Derived Waste Handling

Liquid waste water (i.e., decontamination water and development water and purge water that is pumped from the wells) will be contained in separate drums and tested for hazardous constituents, to determine whether it can be disposed of to the ground at the Property in accordance with applicable regulations, or must be removed from the Property.

All soil cuttings and rock cuttings created during the drilling shall be evaluated using analytical data to determine if they are regulated waste, and temporarily stored in drums on-site, transported to a licensed offsite disposal location, or deposited/discharged on-site. Additional analysis will be performed to fully characterize the wastes in accordance with applicable regulations.

18. EMERGENCY RESPONSE CONTINGENCY PLAN

18.1. Pre-Emergency Planning

The purpose of the emergency response contingency plan is to ensure that in the event of an emergency, personnel on the Site will have the information and understanding in place to institute a reasoned and rational response to the emergency without unnecessary delay. Potential emergencies cover a broad range of occurrences from acute (sudden onset) illness or injury, to fire, explosion, or severe weather conditions. They may be complicated by leaks or by the release of contaminants.

Minimum equipment that will be required on site includes a 20-pound ABC dry chemical fire extinguisher, a first aid kit, and a portable eye wash station. The fire extinguisher, first aid kit and portable eye wash station shall be kept in accessible locations adjacent to the work area.

18.1.1. Employee Training

Employees must be instructed in the site-specific aspects of emergency evacuation. On-site refresher or update training is required anytime escape routes or procedures are modified or personnel assignments are changed.

18.1.2. Alarm Systems/Emergency Signals

An emergency communication system must be in effect at all sites. The most simple and effective emergency communication system in many situations will be direct verbal communications. Each site must be assessed at the time of initial site activity and periodically as the work progresses. Verbal communications must be supplemented anytime voices cannot be clearly perceived above ambient noise levels (i.e., noise from heavy equipment; drilling rigs, backhoes, etc.) and anytime a clear line-of-sight cannot be easily maintained amongst all personnel because of distance, terrain or other obstructions.

An air horn and vehicle horns will be used in the event of an emergency. Three blasts on the horn will indicate that all on-site personnel are to immediately proceed to the primary emergency muster location (designated during the morning health and safety meeting each day based upon the work locations, wind direction, and other pertinent factors).

Verbal communications will be adequate to warn employees of hazards associated with the immediate work area. Telephone facilities are not available in the immediate work areas. Traditional cell phones or other appropriate communications equipment, such as walkie-talkies, will be used to facilitate contact between the team and the USACE on-site representative and/or the local emergency response personnel in the event of an emergency.

18.1.3. Escape Routes and Procedures

The escape route from the work area is via Perimeter Road to Davisville Road.

18.2. Personnel Responsibilities

Emergency response contingency plans can be effective only if the appropriate personnel understand their roles during the emergency, the lines of authority, the training they have received, and the necessity for clear communication.

18.2.1. General Roles and Responsibilities

The role of all site personnel in the event of an emergency is to: communicate to the SSO that an emergency exists; respond appropriately to the particular emergency signal; evacuate and secure the work area or emergency area; conduct the appropriate decontamination activities; render first aid; and call the emergency response services (e.g., the local or regional emergency medical service, fire department, or other specialized personnel or services).

Site personnel shall not engage in firefighting (beyond the use of on-site fire equipment such as extinguishers) or hazardous materials cleanup (beyond clean-up of minor spills or collection of contaminated PPE, etc.). A minor spill is defined as less than one gallon.

Each morning, the SSO will designate a primary and alternative muster location to be used in the event of an emergency. The muster locations will be described during the morning health and safety meeting based upon the work locations, wind direction, and other pertinent factors.

18.2.2. Specific Personnel Responsibilities

Individual personnel associated with site operations, and who will be responsible for maintaining and implementing the emergency response contingency plan, are summarized below.

PM: Responsible for insuring that the features of the emergency response contingency plan are in place, personnel are appropriately trained, and required health safety, and spill response equipment are maintained on the site. In the event the SSO is not able to direct emergency responses the responsibility will fall on the PM. Under those circumstances, the PM will assume the responsibility for evacuation and other emergency actions.

Corporate Health and Safety Officer: Responsible for the overall health and safety of the personnel conducting work associated with a site, compliance with appropriate regulations, review of the emergency response contingency plan to ensure its compliance with all appropriate regulations and company policy, and to review the adequacy of any emergency response and make any corrective measures that may be necessary

Site Technicians and Contractors: Responsible for notifying the SSO immediately upon an emergency, and responding to an emergency situation as described in this plan. The initial response may be required if the SSO is not present on the site.

Site Safety Officer: The SSO is responsible for the initial response to an emergency situation. These duties include the protection of employees, evacuating and securing the work area, ensuring the protection of the public and the environment, ensuring that appropriate decontamination procedures are implemented on all personnel, determining the cause of the incident, recommending changes to prevent its reoccurrence, upgrading or downgrading the level of Personal Protective Equipment, and notification of the appropriate Federal, State and local agencies.

18.2.3. Rescue and Medical Duty Assignments

The phone numbers of the police and fire departments, ambulance service, local hospital, and Stone representatives are provided in the emergency reference sheet located at the end of this APP/SSHP/AHA (before Attachment A).

In the event an injury or illness requires more than first aid treatment, the SSO or his designee will accompany the injured person to the medical facility and will remain with the person until release or admittance is

determined. The escort will relay all appropriate medical information to the PM and the Corporate Health and Safety Officer.

18.2.4. Designation of Responsible Parties

The SSO is responsible for initiating emergency response. In the event the SSO cannot fulfill this duty, the alternate SSO will take charge. All personnel on site are responsible for knowing the escape route from the site and where to assemble after evacuation.

18.2.5. Employee Accounting Method

The SSO is responsible for identifying all team personnel on-site at all times. On small, short duration jobs such as this, this can be done informally as long as accurate accounting (i.e. via headcount) is possible.

18.3. Communication

Every effort shall be made during an emergency to communicate in clear and concise terms. Notice of an emergency shall be communicated to site personnel by a signal from a hand held compressed gas horn. The specific emergency communication signals shall be established by the Site Safety Officer, who will also be responsible for providing the means, such as a compressed gas or vehicle horn, for making this communication. Before work begins, the emergency communication signals shall be made clear to all site workers.

18.4. Emergency Procedures

Personnel shall be familiar with the emergency procedures described below in case of emergencies such as fire, explosion, spills, or leaks. Individuals shall be familiar with their responsibilities in case evacuation of the Site is necessary.

18.4.1. Fire or Explosion

In the event of a fire or explosion, call the Fire Department immediately. Upon arrival of the Fire Department, advise the commanding officer of the location, nature, and identification of hazardous materials on-site. Only trained experienced fire fighters should attempt to extinguish burning materials on site. Site personnel should not attempt to fight any fire, unless properly trained and equipped to do so.

18.4.2. Penetration of Subsurface Containers

It is possible, although highly unlikely, that a drum or similar container was buried on-sit in the past, and could be penetrated during drilling. Symptoms of such an event could include:

- Sudden dropping of the drill string
- Expulsion of noxious vapors from the drill string or ground
- The presence of non-aqueous phase liquids on the drill string or in the hole.

The initial responses to one of these events will be:

- Turn off the drill rig
- Monitor air quality with a PID
- Clear the exclusion zone of all personnel and secure the area with yellow hazard tape

- Notify the FOL.

If the PID readings warrant, or if releases of vapors or liquids continue, the event should be treated similar to a spill, the release contained, and the area evacuated as necessary to protect human health and the environment (see Sections below).

After the area is secure, immediately contact Army Corps of Engineers Project Manager (Casey Haskell: 978-318-8398, cell 978-501-4157).

The State of Rhode Island requires all spills to be reported, so after informing the USACE PM, spills should be reported to the Rhode Island Department of Environmental Management (RIDEM) at 401-222-1360 immediately. Additionally, hazardous waste spills must be reported in writing to RIDEM within 15 days.

18.4.3. Spill or Leaks of Hazardous Materials

18.4.3.1. Scope

This section addresses the potential for spillage/accidental release of containerized liquids that potentially will be present on site. The materials susceptible to spillage that may be present on site are as follows: gasoline, diesel, hydraulic fluid, lubricants, and water in temporary holding tanks.

18.4.3.2. Preventative Measures

All containerized liquids shall be stored in a designated area, isolated from vehicular traffic and protected from the effects of weather. Measures to prevent spills or leaks include:

- all containers shall be labeled as to their contents and principal hazards, in accordance with 29 CFR 1910.120;
- a file of Material Safety Data Sheets (MSDS) shall be maintained by the SSO for all materials introduced to the site;
- flammable or combustible liquids shall be stored separately, and the area shall be placarded to warn of their presence; and
- a supply of specially designed sorbent pads and "minibooms" will be maintained on site for use in spill responses, along with drums suitable for the recovery of used sorbents and contaminated soil.

18.4.3.3. Spill Response Procedures

Training: During the initial on-site health and safety briefing, employees will be informed of the potential hazards of the materials with which they work. The following spill procedures will also be presented at that time. Only those employees trained in accordance with 29 CFR 1910.120 will assist in the clean-up procedure.

Initial response: Upon observing a spill or leakage from a container, employees will immediately step back to a safe distance and direct other employees away from the spill. Initial response actions will include the following:

- Sources of ignition within 100 feet of the spill (including vehicle engines) will be de-energized;
- Inside buildings any exhaust fans will be turned on and overhead doors opened.
- All combustible materials (such as wood, paper, oil, etc.) will be removed from the spill area.

The PM and SSO will be advised immediately of the location, size, and nature of the spill, including the identity of the spilled material (since all containers brought to the Site should be labeled, the spilling of unidentified liquids is not anticipated to be an issue);

Employees who have had contact with the spilled material will be decontaminated as needed.

Planning for Containment/Cleanup: As conditions dictate, the PM or SSO will determine the appropriate follow-up responses. These may include any combination of: initiating containment/clean-up, requesting outside assistance, and notifying affected authorities. The SSO will be notified of the occurrence of any spill in a timely manner.

The SSO will consult the MSDS for the spilled material to determine the specific approach to containing the spill, including the personal protective equipment to be worn during response activities.

The SSO will conduct air monitoring appropriate to the nature of the spilled material, to quantify exposure and detect flammable atmospheres and specify the required PPE for containment/cleanup activities.

Containment/Clean-up: The following are examples of specific measures that may be used (in combination when necessary) to respond to a spill.

- Upright or rotate containers to stop the flow of liquids. This step may be accomplished as soon as the spill or leak occurs, providing it is safe to do so;
- Sorbent pads, booms, or adjacent soil may be used to dike or berm materials subject to flow and to solidify liquids;
- Collect used/saturated sorbent pads, soil or booms in drums;
- Collect contaminated tools and equipment for subsequent cleaning or disposal.

18.4.3.4. Restoration and Salvage of Spill Control Items

The method of disposal of the spill-affected materials will depend on the identity and characteristics of the spilled material. The specific method of disposal, along with any post clean-up verification sampling or restoration procedures, will be determined by the SSO through consultation with the Health and Safety Officer.

Generally, if spill-affected materials such as PPE, sorbent pads and booms, contaminated soil or collected spillage, are determined or suspected as being hazardous waste, they will be collected and sealed in drums that will be appropriately labeled and shipped off-site as a RCRA hazardous waste.

18.4.3.5. Notification

After the area is secure, immediately contact Army Corps of Engineers Project Manager (Casey Haskell: 978-318-8398).

The RIDEM requires all spills to be reported, so after informing the USACE PM, all spills meeting the criteria in DEM-DSR-01-93, Section 5.00 must be reported to RIDEM immediately at 401-222-1360. Additionally, hazardous waste spills must be reported in writing to the DEP within 15 days on the form provided in Attachment H. A written short term response report documenting the measures taken to control and remediate the spill must be filed with RIDEM within 30-days of the release.

18.4.4. Evacuation

Evacuation routes shall be determined at the beginning of each day's work. In an emergency, begin evacuation immediately. Do not delay and let the situation become more dangerous.

Notice to evacuate will be by a continuous blast on an air powered horn or other site-specific audible alarm. Stay upwind of vapors and smoke. Stay upgradient of spills.

If personnel are in an exclusion or decontamination zone at the start of an emergency, they should exit through a decontamination corridor whenever possible. If evacuation cannot be done through a decontamination corridor, site personnel should go to a safe location and remove contaminated clothing there or leave it near the exclusion zone.

19. MODIFICATION OR CHANGES TO THE APP/SSHP/AHA

19.1. Management of Change – Activity Hazard Analysis

The procedures in this APP/SSHP/AHA have been developed based on previous investigations conducted at the Site and the proposed scope of work. Every effort has been made to address the chemical hazards that may be encountered during the implementation of the proposed investigation. Similarly, this document also discusses the physical hazards associated with the proposed activities. However, unanticipated site-specific conditions or situations may occur during the implementation of this project. Also, the Stone and/or Stone contractor (s) may elect to perform certain tasks in a manner that is different from what was originally intended due to a change in field conditions. As such, this APP/SSHP/AHA must be considered a working document that is subject to change to meet the needs of this dynamic project.

Stone and its selected contractor(s) will complete a Activity Hazard Analysis (AHA) when new tasks or different investigative techniques not addressed in the APP/SSHP/AHA are proposed. The use of new techniques will be reviewed and if new hazards are associated with the proposed changes, they will be documented on the AHA form. An effective control measure must also be identified for each new hazard. AHA forms will be reviewed by the SSO prior to being implemented. Once approved, the AHAs will be reviewed with all field staff during the daily safety meeting.

Should significant information become available regarding potential on-site hazards, it may be necessary to modify this APP/SSHP/AHA. All proposed modifications to this APP/SSHP/AHA must be reviewed and approved by the Team Health and Safety Officers before such modifications are implemented. Any significant modifications must be incorporated into the written documentation as addenda and the APP/SSHP/AHA must be reissued. The PM(s) will ensure that all personnel covered by this APP/SSHP/AHA receive copies of all issued addenda. Sign-off forms will accompany each addendum and must be signed by all personnel covered by the addendum. Sign-off forms will be submitted to the PM(s). The APP/SSHP/AHA addenda should be distributed during the daily safety meeting so that they can be reviewed and discussed. Attendance forms will be collected during the meeting.

EMERGENCY REFERENCES

Ambulance/Fire/Police: 911

Medical Services: 401-737-7000
Kent County Memorial Hospital,
455 Tollgate Road, Warwick, RI 02886

Directions to Hospital (See Figure 2)

From PERIMETER RD.

Turn RIGHT onto DAVISVILLE RD.

At the traffic circle, take the 3rd EXIT onto the RI-403 W ramp.

Keep RIGHT at the fork, follow signs for RI-4 N/ I-95/ Providence and merge onto RI-4 N.

RI-4 N becomes I-95 N.

Take EXIT 10 for RI-117 toward Warwick/ Warwick W

Turn RIGHT onto CENTERVILLE RD/ RI-117 E.

Turn LEFT onto TOLL GATE RD/ RI-115 W.

End at 455 Toll Gate Rd Warwick, RI 02886

On Site Telephone: Portable phones will be carried by Field Staff

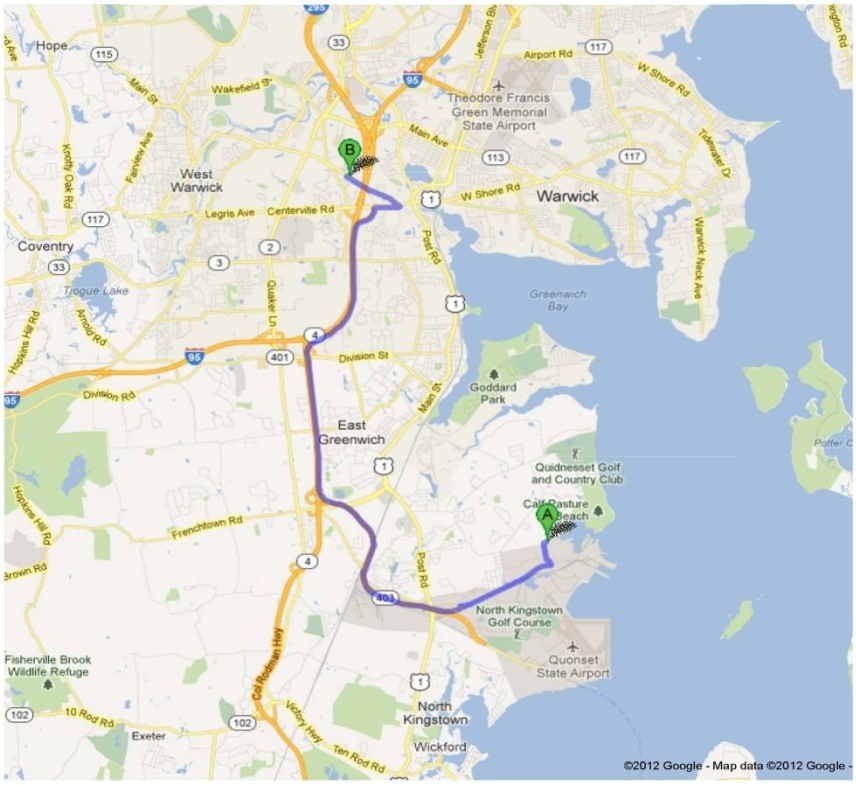
Project Team Contacts:

Title	SSO	Name, Firm	Phone Number	Cell Number	Email
Program Manager, Executive Management	X	Seth Pitkin	802-229-2192	802-249-8700	spitkin@stone-env.com
Project Manager	X	Joseph Schmidl	802-229-5377	802-477-3126	jschmidl@stone-env.com
Site Safety Officer	X	Lee Rosberg	802-229-5378	802-309-1629	lrosberg@stone-env.com
Corporate Health and Safety Officer		Kim Watson	802-299-2196	802-249-7753	kwatson@stone-env.com

Perimeter Rd, North Kingstown, RI 02852 to Kent Hospital - Google Maps Page 1 of 2

Google

Directions to Kent Hospital
455 Toll Gate Road, Warwick, RI 02886 - (401) 737-7000
11.7 mi – about **18 mins**



http://maps.google.com/maps?saddr=Perimeter+Road,+North+Kingstown,+RI&daddr=Ke... 9/18/2012

Figure 2: Route to Hospital

ATTACHMENTS

ATTACHMENT A: HEALTH AND SAFETY PLAN RECEIPT AND ACCEPTANCE FORM

Health and Safety Plan
Receipt and Acceptance Form
Nike Battery PR-58 Site
North Kingstown, Rhode Island

I have received a copy of the Health and Safety Plan prepared for the above referenced site, I have read and understand its content and I agree that I will abide by its requirements.

Name	Signature	Company	Date

ATTACHMENT B: ACTIVITY HAZARD ANALYSIS FORMS

Activity Hazard Analysis (AHA) – 01 Feasibility Study Supplemental Investigation

Task: Site Reconnaissance, Subsurface Utility Clearance, Surveying	**Stone Project Number:** 102305-R/03	
Minimum Personal Protective Equipment (PPE): Level D PPE (Long pants, shirts with minimum 4" sleeve, steel toe boots, safety glasses, hard hat for overhead hazards, leather work gloves, and hearing protection, as required)	**Location:** Nike Battery Site PR-58 North Kingstown, RI	
	Analysis Approved by: Kim B. Watson, Corporate Health and Safety	**Date:** September 2012

Activity	Potential Hazards	Recommended Controls
Perform non-intrusive site activities, including Site Reconnaissance, Subsurface Utility Clearance, and Surveying	**Chemical Hazards:** Potential for inhalation and/or contact exposure to chlorinated compounds from contaminated groundwater	**Chemical Hazards**: • Use of PPE during intrusive activities in source areas (modified Level D) to limit contact risk. Air monitoring of breathing zone with photoionization detector for the presence of volatile organic compounds (e.g., trichloroethylene (TCE) and tetrachloroethene (PCE)). Proper management of contaminated groundwater and decontamination water.
	Potential for exposure to chemicals brought on-site.	• MSDS for all chemical brought on-site shall be provided to the SSO. Conduct a utility locate to identify the location of underground utilities in boring locations and complete any required dig permits
	Physical Hazards: Slip, trip, fall hazards.	**Physical Hazards:** • Employ safe work practices and good housekeeping: keep work area free of slippery walking surfaces; keep tools and materials from underfoot; maintain three points of contact when climbing on/off drilling equipment. Apply sand to any ice covered surfaces within work areas prior to conducting field activities or utilize traction devices (e.g., ice creepers).
	Thermal Stressors (i.e. heat stress, cold stress)	• Employees will have appropriate clothing for variable weather • Use of long sleeves or application of sunscreen with a high sun protection factor (SPF) on exposed skin encouraged • Employees will take breaks and drink plenty of fluids to prevent heat stress • Warming breaks will be permitted as necessary to prevent cold stress
	Inclement weather (Thunderstorms and tornadoes)	• Halt activities immediately and take cover during thunderstorm or tornado warnings, shelter in a building if possible, stay away from windows • If outdoors, stay close to the ground • Listen to radio or television announcements for pending weather information • Do not try to outrun a tornado on foot or in a vehicle
	Biological hazards (insects, spiders, etc.)	• Workers will inspect the work area carefully and avoid placing hands and feet into concealed areas Look in direction of travel for biological hazards to avoid

AHA – 01 (Concluded)

Activity	Potential Hazards	Recommended Controls
	Electrical hazards	• None
	Water hazard, working near or on water - immersion in water - drowning	• Personnel will avoid waterbodies.
Equipment Used	**Inspection Requirements**	**Training Requirements**
Vehicles and site equipment Pickup trucks or vans	Inspect all equipment prior to use; refer to manufacturers inspection requirements, if appropriate	Valid operators license for the class of vehicle.
Geophysical instruments; Total Station or Autolevel, Stadia rod.	Calibrate and use instruments/equipment in accordance with manufacturers recommendation	Experience in groundwater quality sampling utilizing low-flow techniques and equipment. Training in related SEI SOPs.
Decontamination containment vessels/structures including containers for purge water from work in source areas wells	Prior to each use, inspect any decontamination containment vessel or structure. Inspect any container prior before depositing any soil/rock cuttings, groundwater or decontamination residuals from work in source areas.	No specific training required.
		General: Review of Site-Specific Health and Safety Plan and Activity Hazard Analyses Hazardous Sites Worker training per 29CFR 1910.120 Initial site/task specific training. Daily Safety Meetings. Review EM 385-1-1 Section 11.A – 11.D.

Activity Hazard Analysis (AHA) – 02 Feasibility Study Supplemental Investigation

Task: Surface Water, Porewater, Sediment, and Monitoring well sample collection		**Stone Project Number:** 102305-R/03	
Minimum Personal Protective Equipment (PPE): Level D PPE (Long pants, shirts with minimum 4" sleeve, steel toe boots, safety glasses, hard hat for overhead hazards, leather work gloves, and hearing protection, as required)		**Location:** Nike Battery Site PR-58 North Kingstown, RI	
		Analysis Approved by: Kim B. Watson, Corporate Health and Safety	**Date:** September 2012

Activity	Potential Hazards	Recommended Controls
Collect surface water, sediment and porewater samples	**Chemical Hazards:** Potential for inhalation and/or contact exposure to chlorinated compounds from contaminated groundwater	**Chemical Hazards**: • Use of PPE during intrusive activities in source areas (modified Level D) to limit contact risk. Air monitoring of breathing zone with photoionization detector for the presence of volatile organic compounds (e.g., trichloroethylene (TCE) and tetrachloroethene (PCE)). Proper management of contaminated groundwater and decontamination water.
	Potential for exposure to chemicals brought on-site.	• MSDS for all chemical brought on-site shall be provided to the SSO. Conduct a utility locate to identify the location of underground utilities in boring locations and complete any required dig permits
	Physical Hazards: Slip, trip, fall hazards.	**Physical Hazards:** • Employ safe work practices and good housekeeping: keep work area free of slippery walking surfaces; keep tools and materials from underfoot; maintain three points of contact when climbing on/off drilling equipment. Apply sand to any ice covered surfaces within work areas prior to conducting field activities or utilize traction devices (e.g., ice creepers).
	Thermal Stressors (i.e. heat stress, cold stress)	• Employees will have appropriate clothing for variable weather • Use of long sleeves or application of sunscreen with a high sun protection factor (SPF) on exposed skin encouraged • Employees will take breaks and drink plenty of fluids to prevent heat stress • Warming breaks will be permitted as necessary to prevent cold stress
	Inclement weather (Thunderstorms and tornadoes)	• Halt activities immediately and take cover during thunderstorm or tornado warnings, shelter in a building if possible, stay away from windows • If outdoors, stay close to the ground • Listen to radio or television announcements for pending weather information • Do not try to outrun a tornado on foot or in a vehicle

AHA – 02 (continued)

Activity	Potential Hazards	Recommended Controls
	Biological hazards (insects, spiders etc.)	• Workers will inspect the work area carefully and avoid placing hands and feet into concealed areas Look in direction of travel for biological hazards to avoid
	Electrical hazards	• Sampling pumps shall be grounded by a multi-conductor cord with identified grounding conductor and multi-contact plug and receptacle. Comply with Section 11.A – 11.D of EM 385-1-1.
	Water hazard, working near or on water - immersion in water - drowning	• Sampling personnel will be accessing Davol Pond – the buddy system or pre-post sampling communication will be used. Personnel will be accessing sampling site with a canoe or skiff. The appropriate PFDs will be on board and in use.
Equipment Used	**Inspection Requirements**	**Training Requirements**
Vehicles and site equipment Pickup trucks or vans Canoe or small boat Portable generator Flexible extension cords Hand tools	Inspect all equipment prior to use; refer to manufacturers inspection requirements, if appropriate. Inspect flexible extension cords daily. Test GFIC circuits prior to use	Valid operator's license for the class of vehicle. Experience in maneuvering small watercraft.
Photoionization detector and water level indicator; sampling equipment including: downhole submersible pump, multi-parameter probe; turbidity meter; pre-preserved sample containers; and sampling tubing.	Inspect and calibrate Photoionization detector (PID) daily when in use to monitor intrusive activities. Calibrate and use instruments/equipment in accordance with manufacturers recommendation	Experience in operating and calibrating a PID. Experience in groundwater quality sampling utilizing low-flow techniques and equipment. Training in related SEI SOPs.
Decontamination containment vessels/structures including containers for purge water from work in source areas wells	Prior to each use, inspect any decontamination containment vessel or structure. Inspect any container prior before depositing any soil/rock cuttings, groundwater or decontamination residuals from work in source areas.	No specific training required.

AHA – 02 (Concluded)

Equipment Used	Inspection Requirements	Training Requirements
		General: Review of Site-Specific Health and Safety Plan and Activity Hazard Analyses Hazardous Sites Worker training per 29CFR 1910.120 Initial site/task specific training. Daily Safety Meetings. Review EM 385-1-1 Section 11.A – 11.D.

Activity Hazard Analysis (AHA) – 03 Feasibility Study Supplemental Investigation

Task: Monitoring Well Sample Collection	**Stone Project Number:** 102305-R/03	
Minimum Personal Protective Equipment (PPE): Level D PPE (Long pants, shirts with minimum 4" sleeve, steel toe boots, safety glasses, hard hat for overhead hazards, leather work gloves, and hearing protection, as required)	**Location:** Nike Battery Site PR-58 North Kingstown, RI	
	Analysis Approved by: Kim B. Watson, Corporate Health and Safety	**Date:** September 2012

Activity	Potential Hazards	Recommended Controls
Collect groundwater samples from monitoring wells	**Chemical Hazards:** Potential for inhalation and/or contact exposure to chlorinated compounds from contaminated groundwater	**Chemical Hazards**: • Use of PPE during intrusive activities in source areas (modified Level D) to limit contact risk. Air monitoring of breathing zone with photoionization detector for the presence of volatile organic compounds (e.g., trichloroethylene (TCE) and tetrachloroethene (PCE)). Proper management of contaminated groundwater and decontamination water.
	Potential for exposure to chemicals brought on-site.	• MSDS for all chemical brought on-site shall be provided to the SSO. Conduct a utility locate to identify the location of underground utilities in boring locations and complete any required dig permits
	Physical Hazards: Slip, trip, fall hazards.	**Physical Hazards:** • Employ safe work practices and good housekeeping: keep work area free of slippery walking surfaces; keep tools and materials from underfoot; maintain three points of contact when climbing on/off drilling equipment. Apply sand to any ice covered surfaces within work areas prior to conducting field activities or utilize traction devices (e.g., ice creepers).
	Thermal Stressors (i.e. heat stress, cold stress)	• Employees will have appropriate clothing for variable weather • Use of long sleeves or application of sunscreen with a high sun protection factor (SPF) on exposed skin encouraged • Employees will take breaks and drink plenty of fluids to prevent heat stress • Warming breaks will be permitted as necessary to prevent cold stress
	Inclement weather (Thunderstorms and tornadoes)	• Halt activities immediately and take cover during thunderstorm or tornado warnings, shelter in a building if possible, stay away from windows • If outdoors, stay close to the ground • Listen to radio or television announcements for pending weather information • Do not try to outrun a tornado on foot or in a vehicle

AHA – 03 (continued)

Activity	Potential Hazards	Recommended Controls
	Biological hazards (insects, spiders etc.)	• Workers will inspect the work area carefully and avoid placing hands and feet into concealed areas Look in direction of travel for biological hazards to avoid
	Electrical hazards	• Sampling pumps shall be grounded by a multi-conductor cord with identified grounding conductor and multi-contact plug and receptacle. Comply with Section 11.A – 11.D of EM 385-1-1.
	Water hazard, working near or on water - immersion in water - drowning	• Sampling personnel will be accessing Davol Pond – the buddy system or pre-post sampling communication will be used. Personnel will be accessing sampling site with a canoe or skiff. The appropriate PFDs will be on board and in use.
Equipment Used	**Inspection Requirements**	**Training Requirements**
Vehicles and site equipment Pickup trucks or vans Canoe or small boat Portable generator Flexible extension cords Hand tools	Inspect all equipment prior to use; refer to manufacturers inspection requirements, if appropriate. Inspect flexible extension cords daily. Test GFIC circuits prior to use	Valid operators license for the class of vehicle. Experience in maneuvering small watercraft.
Photoionization detector and water level indicator; sampling equipment including: downhole submersible pump, multi-parameter probe; turbidity meter; pre-preserved sample containers; and sampling tubing.	Inspect and calibrate Photoionization detector (PID) daily when in use to monitor intrusive activities. Calibrate and use instruments/equipment in accordance with manufacturers recommendation	Experience in operating and calibrating a PID. Experience in groundwater quality sampling utilizing low-flow techniques and equipment. Training in related SEI SOPs.
Decontamination containment vessels/structures including containers for purge water from work in source areas wells	Prior to each use, inspect any decontamination containment vessel or structure. Inspect any container prior before depositing any soil/rock cuttings, groundwater or decontamination residuals from work in source areas.	No specific training required.

AHA – 03 (Concluded)

Equipment Used	Inspection Requirements	Training Requirements
		General: Review of Site-Specific Health and Safety Plan and Activity Hazard Analyses Hazardous Sites Worker training per 29CFR 1910.120 Initial site/task specific training. Daily Safety Meetings. Review EM 385-1-1 Section 11.A – 11.D.

Activity Hazard Analysis (AHA) – 04 Feasibility Study Supplemental Investigation

Task: Drinking Water Sample Collection		**Stone Project Number:** 102305-R/03	
Minimum Personal Protective Equipment (PPE): Level D PPE (Long pants, shirts with minimum 4" sleeve, steel toe boots, safety glasses, hard hat for overhead hazards, leather work gloves, and hearing protection, as required)		**Location:** Nike Battery Site PR-58 North Kingstown, RI	
		Analysis Approved by: Kim B. Watson, Corporate Health and Safety	**Date:** September 2012

Activity	Potential Hazards	Recommended Controls
Collect drinking water samples from residential wells, at the tap	**Chemical Hazards:** Potential for inhalation and/or contact exposure to chlorinated compounds from contaminated groundwater	**Chemical Hazards**: • Use of PPE during intrusive activities in source areas (modified Level D) to limit contact risk. Air monitoring of breathing zone with photoionization detector for the presence of volatile organic compounds (e.g., trichloroethylene (TCE) and tetrachloroethene (PCE)). Proper management of contaminated groundwater and decontamination water.
	Potential for exposure to chemicals brought on-site.	• MSDS for all chemical brought on-site shall be provided to the SSO. Conduct a utility locate to identify the location of underground utilities in boring locations and complete any required dig permits
	Physical Hazards: Slip, trip, fall hazards.	**Physical Hazards:** • Employ safe work practices and good housekeeping: keep work area free of slippery walking surfaces; keep tools and materials from underfoot; maintain three points of contact when climbing on/off drilling equipment. Apply sand to any ice covered surfaces within work areas prior to conducting field activities or utilize traction devices (e.g., ice creepers).
	Thermal Stressors (i.e. heat stress, cold stress)	• Employees will have appropriate clothing for variable weather • Use of long sleeves or application of sunscreen with a high sun protection factor (SPF) on exposed skin encouraged • Employees will take breaks and drink plenty of fluids to prevent heat stress • Warming breaks will be permitted as necessary to prevent cold stress
	Inclement weather (Thunderstorms and tornadoes)	• Halt activities immediately and take cover during thunderstorm or tornado warnings, shelter in a building if possible, stay away from windows • If outdoors, stay close to the ground • Listen to radio or television announcements for pending weather information • Do not try to outrun a tornado on foot or in a vehicle

Activity	Potential Hazards	Recommended Controls
	Biological hazards (insects, spiders etc.)	• Workers will inspect the work area carefully and avoid placing hands and feet into concealed areas Look in direction of travel for biological hazards to avoid
	Electrical hazards	• Sampling pumps shall be grounded by a multi-conductor cord with identified grounding conductor and multi-contact plug and receptacle. Comply with Section 11.A – 11.D of EM 385-1-1.
	Water hazard, working near or on water - immersion in water - drowning	• Sampling personnel will be accessing Davol Pond – the buddy system or pre-post sampling communication will be used. Personnel will be accessing sampling site with a canoe or skiff. The appropriate PFDs will be on board and in use.
Equipment Used	**Inspection Requirements**	**Training Requirements**
Vehicles and site equipment Pickup trucks or vans Canoe or small boat Portable generator Flexible extension cords Hand tools	Inspect all equipment prior to use; refer to manufacturers inspection requirements, if appropriate. Inspect flexible extension cords daily. Test GFIC circuits prior to use	Valid operators license for the class of vehicle. Experience in maneuvering small watercraft.
Photoionization detector and water level indicator; sampling equipment including: downhole submersible pump, multi-parameter probe; turbidity meter; pre-preserved sample containers; and sampling tubing.	Inspect and calibrate Photoionization detector (PID) daily when in use to monitor intrusive activities. Calibrate and use instruments/equipment in accordance with manufacturers recommendation	Experience in operating and calibrating a PID. Experience in groundwater quality sampling utilizing low-flow techniques and equipment. Training in related SEI SOPs.
Decontamination containment vessels/structures including containers for purge water from work in source areas wells	Prior to each use, inspect any decontamination containment vessel or structure. Inspect any container prior before depositing any soil/rock cuttings, groundwater or decontamination residuals from work in source areas.	No specific training required.

AHA – 04 (Concluded)

Equipment Used	Inspection Requirements	Training Requirements
		General: Review of Site-Specific Health and Safety Plan and Activity Hazard Analyses Hazardous Sites Worker training per 29CFR 1910.120 Initial site/task specific training. Daily Safety Meetings. Review EM 385-1-1 Section 11.A – 11.D.

Activity Hazard Analysis (AHA) – 05 Feasibility Study Supplemental Investigation

Task: Overburden Soil Sampling and Well Drilling	**Stone Project Number:** 102305-R/03	
Minimum Personal Protective Equipment (PPE): Level D PPE (Long pants, shirts with minimum 4" sleeve, steel toe boots, safety glasses, hard hat for overhead hazards, leather work gloves, and hearing protection, as required)	**Location:** Nike Battery Site PR-58 North Kingstown, RI	
	Analysis Approved by: Kim B. Watson, Corporate Health and Safety	**Date:** September 2012

Activity	Potential Hazards	Recommended Controls
Positioning and operation of drill rigs and associated tools/equipment. Obtaining/handling soil and rock cutting samples Decontamination of drill rig(s), drill tools, and associated equipment. Handling and management of soil/rock cuttings and drilling fluids.	**Chemical Hazards**: Potential for inhalation and/or contact exposure to chlorinated compounds from contaminated groundwater and/or soil/rock.	**Chemical Hazards**: • Use of PPE during intrusive activities in source areas (modified Level D) to limit contact risk. Air monitoring of breathing zone with photoionization detector for the presence of volatile organic compounds (e.g., trichloroethylene (TCE) and tetrachloroethene (PCE)). Proper management of rock/soil cuttings, contaminated groundwater and decontamination solids/water.
	Potential for exposure to chemicals brought on-site.	• MSDS for all chemicals brought on-site shall be provided to the Site Safety Officer.
	Physical Hazards: Struck by/entanglement in and/or pinched between equipment while positioning and operating drill rig, drilling tools and associated equipment.	**Physical Hazards**: • Employ safe work practices; make eye contact with operator if need to enter work area; restrict work area to essential personnel. Wear specified PPE including hard hats, eye protection and safety toed work boots. Utilize procedures outlined in the SEI Drilling Safety Program.
	Exposure to excessive noise levels. Exposure to Eye injury hazards	• Utilize hearing protection against excessive noise levels and wear eye protection.
	Contact with overhead/underground utilities	• Maintain required isolation distances from overhead power lines. Utilize Dig Safe to identify any underground utilities and to clear each drilling location. Determine that no known underground utilities exist in the drilling locations
	Thermal Stressors (i.e. heat stress, cold stress)	• Employees will have appropriate clothing for variable weather • Use of long sleeves or application of sunscreen with a high sun protection factor (SPF) on exposed skin encouraged • Employees will take breaks and drink plenty of fluids to prevent heat stress • Warming breaks will be permitted as necessary to prevent cold stress

Activity	Potential Hazards	Recommended Controls
	Back Injury from Materials Handling	• Use proper lifting techniques • Loads greater than 50 pounds require assistance or mechanical equipment • Prior to lifting, check the load for jagged or sharp edges • Avoid torso twisting motions while handling or moving loads
	Slip, trip, fall hazards.	• Employ safe work practices and good housekeeping: keep work area free of slippery walking surfaces; keep tools and materials from underfoot; maintain three points of contact when climbing on/off drilling equipment. Apply sand to any ice covered surfaces within work areas prior to conducting field activities or utilize traction devices (e.g., ice creepers).
	Inclement weather (Thunderstorms and tornadoes)	• Halt activities immediately and take cover during thunderstorm or tornado warnings, shelter in a building if possible, stay away from windows • If outdoors, stay close to the ground • Listen to radio or television announcements for pending weather information • Do not try to outrun a tornado on foot or in a vehicle
	Biological hazards (insects, spiders etc.)	• Workers will inspect the work area carefully and avoid placing hands and feet into concealed areas Look in direction of travel for biological hazards to avoid
	Contact with hot water or high-pressure sprayer	• Utilize eye protection and leather or heavy rubber (Petroflex or equivalent) gloves. Maintain a safe distance from the discharge of the spray wand. Never point the wand at another person nor use for personal decontamination.
	Compressed gas can be driven into the flesh or eyes.	• Utilize eye protection. Pneumatic impact tools and hoses should never be pointed at a person. Before hoses are disconnected, the air supply must be turned off at the control valve and the pressure bled off.
Equipment Used	**Inspection Requirements**	**Training Requirements**
Vehicle and site equipment Drill rigs Pickup trucks or vans Steam or high temperature pressure washer Air compressor Portable generator Hand tools	Inspect drill rig before initial use in accordance with EM 385-1-1 (16.M.02) and per The SEI's Drilling Safety Program or Subcontractors equivalent. Inspect all equipment prior to use; refer to manufacturers inspection requirements, if appropriate.	Valid operators license for the class of vehicle. Experience in drill rig operation.

Equipment Used	Inspection Requirements	Training Requirements
Photoionization detector	Inspect and calibrate Photoionization detector (PID) daily when in use to monitor intrusive activities.	Experience in operating and calibrating a PID.
Decontamination containment vessels/structures: container for decontamination residuals from work in source areas	Prior to each use, inspect any decontamination containment vessel or structure. Inspect any container prior before depositing any soil/rock cuttings, groundwater or decontamination residuals from work in source areas.	No specific training required.
Air compressor or compressed inert gas.	Prior to each use, inspect all air hoses, couplings and connections to determine if leakage or other damage exists. All pipes, hoses, and fittings must have a rating of the maximum pressure of the compressor or tank, should be kept clean, and hose ends must be secured to prevent whipping if an accidental cut or break occurs.	No specific training required. Be familiar with operator's manual.
		General: Review of Site-Specific Health and Safety Plan and Activity Hazard Analyses Hazardous Sites Worker training per 29CFR 1910.120 Initial site/task specific training. Daily Safety Meetings.

Activity Hazard Analysis (AHA) – 06 Feasibility Study Supplemental Investigation

Task: Chemical Analysis in Mobile Laboratory		**Stone Project Number:** 102305-R/03	
Minimum Personal Protective Equipment (PPE): Level D PPE (Long pants, shirts with minimum 4" sleeve, steel toe boots, safety glasses, hard hat for overhead hazards, leather work gloves, and hearing protection, as required)		**Location:** Nike Battery Site PR-58 North Kingstown, RI	
		Analysis Approved by: Kim B. Watson, Corporate Health and Safety	**Date:** September 2012

Activity	Potential Hazards	Recommended Controls
Preparation of VOA vials for soil sampling. Receiving, handling, storing and analysis of water and soil samples.	**Chemical Hazards:** Potential for inhalation and/or contact exposure to chlorinated compounds from contaminated groundwater	**Chemical Hazards**: • Use of PPE during intrusive activities in source areas (modified Level D) to limit contact risk. Air monitoring of breathing zone with photoionization detector for the presence of volatile organic compounds (e.g., trichloroethylene (TCE) and tetrachloroethene (PCE)). Proper management of contaminated groundwater and decontamination water.
	Potential for exposure to chemicals brought on-site.	• MSDS for all chemical brought on-site shall be provided to the SSO. Conduct a utility locate to identify the location of underground utilities in boring locations and complete any required dig permits
	Physical Hazards: Compressed gas can be driven into the flesh or eyes.	**Physical Hazards:** • Utilize eye protection. Pneumatic impact tools and hoses should never be pointed at a person. Before hoses are disconnected, the air supply must be turned off at the control valve and the pressure bled off.
Equipment Used	**Inspection Requirements**	**Training Requirements**
Gas Chromatograph (GC) with FID detector and Mass Spectrometer Detector (GC/MSD). Laboratory supplies: volumetric syringes, stirring plates, beakers. Hydrogen, helium and purified air gas cylinders.	Inspect all equipment prior to use; refer to manufacturers inspection requirements, if appropriate. Inspect and calibrate GC/GC/MS daily as required in the standard method. Inspect laboratory equipment and remove defective devices from service.	1) Corporate and Laboratory H&S training. NELAC training and training on the Laboratory Quality Manual And reference to reading and understanding the Standard Operating Procedures 2) Experience in operating and calibrating a GC/GC/MSD.
		General: Review of Site-Specific Health and Safety Plan and Activity Hazard Analyses Hazardous Sites Worker training per 29CFR 1910.120 Initial site/task specific training. Daily Safety Meetings.

Activity Hazard Analysis (AHA) – 07 Feasibility Study Supplemental Investigation

Task: Install and Develop Monitoring Wells	**Stone Project Number:** 102305-R/03	
Minimum Personal Protective Equipment (PPE): Level D PPE (Long pants, shirts with minimum 4" sleeve, steel toe boots, safety glasses, hard hat for overhead hazards, leather work gloves, and hearing protection, as required)	**Location:** Nike Battery Site PR-58 North Kingstown, RI	
	Analysis Approved by: Kim B. Watson, Corporate Health and Safety	**Date:** September 2012

Activity	Potential Hazards	Recommended Controls
Installation and development of monitoring wells. Decontamination of drill rig(s), drill tools, and associated equipment. Handling and management of well development water.	**Chemical Hazards**: Potential for inhalation and/or contact exposure to chlorinated compounds from contaminated groundwater and/or soil/rock.	**Chemical Hazards**: • Use of PPE during intrusive activities in source areas (modified Level D) to limit contact risk. Air monitoring of breathing zone with photoionization detector for the presence of volatile organic compounds (e.g., trichloroethylene (TCE) and tetrachloroethene (PCE)). Proper management of rock/soil cuttings, contaminated groundwater and decontamination solids/water.
	Potential for exposure to chemicals brought on-site.	• MSDS for all chemicals brought on-site shall be provided to the Site Safety Officer.
	Physical Hazards: Struck by/entanglement in and/or pinched between equipment while positioning and operating drill rig, drilling tools and associated equipment.	**Physical Hazards**: • Employ safe work practices; make eye contact with operator if need to enter work area; restrict work area to essential personnel. Wear specified PPE including hard hats, eye protection and safety toed work boots. Utilize procedures outlined in the SEI Drilling Safety Program.
	Exposure to excessive noise levels. Exposure to Eye injury hazards	• Utilize hearing protection against excessive noise levels and wear eye protection.
	Contact with overhead/underground utilities	• Maintain required isolation distances from overhead power lines. Utilize Dig Safe to identify any underground utilities and to clear each drilling location. Determine that no known underground utilities exist in the drilling locations
	Thermal Stressors (i.e. heat stress, cold stress)	• Employees will have appropriate clothing for variable weather • Use of long sleeves or application of sunscreen with a high sun protection factor (SPF) on exposed skin encouraged • Employees will take breaks and drink plenty of fluids to prevent heat stress • Warming breaks will be permitted as necessary to prevent cold stress

AHA – 07 (Continued)

Activity	Potential Hazards	Recommended Controls
	Back Injury from Materials Handling	• Use proper lifting techniques • Loads greater than 50 pounds require assistance or mechanical equipment • Prior to lifting, check the load for jagged or sharp edges • Avoid torso twisting motions while handling or moving loads
	Slip, trip, fall hazards.	• Employ safe work practices and good housekeeping: keep work area free of slippery walking surfaces; keep tools and materials from underfoot; maintain three points of contact when climbing on/off drilling equipment. Apply sand to any ice covered surfaces within work areas prior to conducting field activities or utilize traction devices (e.g., ice creepers).
	Inclement weather (Thunderstorms and tornadoes)	• Halt activities immediately and take cover during thunderstorm or tornado warnings, shelter in a building if possible, stay away from windows • If outdoors, stay close to the ground • Listen to radio or television announcements for pending weather information • Do not try to outrun a tornado on foot or in a vehicle
	Biological hazards (insects, spiders etc.)	• Workers will inspect the work area carefully and avoid placing hands and feet into concealed areas Look in direction of travel for biological hazards to avoid
	Contact with hot water or high-pressure sprayer	• Utilize eye protection and leather or heavy rubber (Petroflex or equivalent) gloves. Maintain a safe distance from the discharge of the spray wand. Never point the wand at another person nor use for personal decontamination.
	Compressed gas can be driven into the flesh or eyes.	• Utilize eye protection. Pneumatic impact tools and hoses should never be pointed at a person. Before hoses are disconnected, the air supply must be turned off at the control valve and the pressure bled off.

Equipment Used	Inspection Requirements	Training Requirements
Vehicle and site equipment Drill rigs Pickup trucks or vans Steam or high temperature pressure washer Air compressor Portable generator Hand tools	Inspect drill rig before initial use in accordance with EM 385-1-1 (16.M.02) and per The SEI's Drilling Safety Program or Subcontractors equivalent. Inspect all equipment prior to use; refer to manufacturers inspection requirements, if appropriate.	Valid operator license for the class of vehicle. Experience in drill rig operation.
Photoionization detector	Inspect and calibrate Photoionization detector (PID) daily when in use to monitor intrusive activities.	Experience in operating and calibrating a PID.
Decontamination containment vessels/structures: container for decontamination residuals from work in source areas	Prior to each use, inspect any decontamination containment vessel or structure. Inspect any container prior before depositing any soil/rock cuttings, groundwater or decontamination residuals from work in source areas.	No specific training required.
Air compressor or compressed inert gas.	Prior to each use, inspect all air hoses, couplings and connections to determine if leakage or other damage exists. All pipes, hoses, and fittings must have a rating of the maximum pressure of the compressor or tank, should be kept clean, and hose ends must be secured to prevent whipping if an accidental cut or break occurs.	No specific training required. Be familiar with operator's manual.
		General: Review of Site-Specific Health and Safety Plan and Activity Hazard Analyses Hazardous Sites Worker training per 29CFR 1910.120 Initial site/task specific training. Daily Safety Meetings.

Activity Hazard Analysis (AHA) – 08 Feasibility Study Supplemental Investigation

Task: Contaminated water treatment and/or disposal; contaminated solids treatment and/or disposal	**Stone Project Number:** 102305-R/03	
Minimum Personal Protective Equipment (PPE): Level D PPE (Long pants, shirts with minimum 4" sleeve, steel toe boots, safety glasses, hard hat for overhead hazards, leather work gloves, and hearing protection, as required)	**Location:** Nike Battery Site PR-58 North Kingstown, RI	
	Analysis Approved by: Kim B. Watson, Corporate Health and Safety	**Date:** September 2012

Activity	Potential Hazards	Recommended Controls
Treating via activated carbon or discharging water to the ground. Positioning and filling drums, containers or tanks for temporary storage or on-site transport and subsequent depositing them onto the ground surface. Handling and management of rock, soil and cuttings, samples, groundwater and decontamination residuals.	**Chemical Hazards**: Potential for inhalation and/or contact exposure to chlorinated compounds from contaminated groundwater and/or solids/soil/rock.	**Chemical Hazards**: • Use of PPE during intrusive activities in source areas (modified Level D) to limit contact risk. Air monitoring of breathing zone with photoionization detector for the presence of volatile organic compounds (e.g., trichloroethylene (TCE) and tetrachloroethene (PCE)). Proper management of rock/soil cuttings, contaminated groundwater and decontamination solids/water.
	Potential for exposure to chemicals brought on-site.	• MSDS for all chemical brought on-site shall be provided to the SSO. Conduct a utility locate to identify the location of underground utilities in boring locations and complete any required dig permits
	Physical Hazards: Struck by/entanglement and/or pinched between while positioning and filling containers.	**Physical Hazards**: • Employ safe work practices; make eye contact with operator if need to enter work area; restrict work area to essential personnel. Wear protective equipment including hard hats, eye protection and safety toed work boots.
	Exposure to excessive noise levels. Exposure to eye injury hazards	• Utilize hearing protection against excessive noise levels and wear eye protection.
	Contact with overhead/underground utilities	• Maintain required isolation distances from overhead power lines.

AHA – 08 (continued)

Activity	Potential Hazard(s)	Recommended Controls
	Back Injury from Materials Handling	• Use proper lifting techniques • Loads greater than 50 pounds require assistance or mechanical equipment • Prior to lifting, check the load for jagged or sharp edges • Avoid torso twisting motions while handling or moving loads
	Slip, trip, fall hazards.	• Employ safe work practices and good housekeeping: keep work area free of slippery walking surfaces; keep tools and materials from underfoot; maintain three points of contact when climbing on/off drilling equipment. Apply sand to any ice covered surfaces within work areas prior to conducting field activities or utilize traction devices (e.g., ice creepers).
	Thermal Stressors (i.e. heat stress, cold stress)	• Employees will have appropriate clothing for variable weather • Use of long sleeves or application of sunscreen with a high sun protection factor (SPF) on exposed skin encouraged • Employees will take breaks and drink plenty of fluids to prevent heat stress • Warming breaks will be permitted as necessary to prevent cold stress
	Inclement weather (Thunderstorms and tornadoes)	• Halt activities immediately and take cover during thunderstorm or tornado warnings, shelter in a building if possible, stay away from windows • If outdoors, stay close to the ground • Listen to radio or television announcements for pending weather information • Do not try to outrun a tornado on foot or in a vehicle
	Biological hazards (insects, spiders etc.)	• Workers will inspect the work area carefully and avoid placing hands and feet into concealed areas Look in direction of travel for biological hazards to avoid

AHA – 08 (concluded)

Equipment Used	Inspection Requirements	Training Requirements
Vehicles and site equipment Excavator, skid steer Pickup trucks or vans Portable tanks, containers, hoses Hand tools	Inspect all equipment prior to use; refer to manufacturers inspection requirements, if appropriate.	Valid operators license for the class of vehicle.
Photoionization detector	Inspect and calibrate Photoionization detector (PID) daily when in use to monitor intrusive activities.	Experience in operating and calibrating a PID.
Dumpster, drums, tanks or containers for temporary storage or on-site transportation of soil/rock cuttings, groundwater and decontamination residuals from work in source areas	Prior to each use, inspect any containment vessel or structure. Inspect any container prior before depositing any soil/rock cuttings, groundwater or decontamination residuals from work in source areas.	No specific training required.
		General: Review of Site-Specific Health and Safety Plan and Activity Hazard Analyses Hazardous Sites Worker training per 29CFR 1910.120 Initial site/task specific training. Daily Safety Meetings.

Activity Hazard Analysis (AHA) – 09 Feasibility Study Supplemental Investigation

Task: Equipment and Personnel Decontamination		**Stone Project Number:** 102305-R/03	
Minimum Personal Protective Equipment (PPE): Level D PPE (Long pants, shirts with minimum 4" sleeve, steel toe boots, safety glasses, hard hat for overhead hazards, leather work gloves, and hearing protection, as required)		**Location:** Nike Battery Site PR-58 North Kingstown, RI	
		Analysis Approved by: Kim B. Watson, Corporate Health and Safety	**Date:** September 2012

Activity	Potential Hazards	Recommended Controls
Decontamination of equipment that comes in contact with contaminated media. Proper management and disposal of PPE. Use of hot-water pressure sprayer for equipment decontamination and the collection, handling and management of decontamination residuals.	**Chemical Hazards:** Potential for inhalation and/or contact exposure to chlorinated compounds from contaminated groundwater	**Chemical Hazards**: • Use of PPE during intrusive activities in source areas (modified Level D) to limit contact risk. Air monitoring of breathing zone with photoionization detector for the presence of volatile organic compounds (e.g., trichloroethylene (TCE) and tetrachloroethene (PCE)). Proper management of contaminated groundwater and decontamination water.
	Potential for exposure to chemicals brought on-site.	• MSDS for all chemical brought on-site shall be provided to the SSO. Conduct a utility locate to identify the location of underground utilities in boring locations and complete any required dig permits
	Physical Hazards: Slip, trip, fall hazards.	**Physical Hazards:** • Employ safe work practices and good housekeeping: keep work area free of slippery walking surfaces; keep tools and materials from underfoot; maintain three points of contact when climbing on/off drilling equipment. Apply sand to any ice covered surfaces within work areas prior to conducting field activities or utilize traction devices (e.g., ice creepers).
	Struck by/entanglement while positioning and operating drill rig, drilling tools, excavator and associated equipment for decontamination.	• Struck by/entanglement while positioning and operating drill rig, drilling tools, excavator and associated equipment for decontamination.
	Exposure to excessive noise levels	• Utilize hearing protection against excessive noise levels.
	Contact with hot water or high-pressure sprayer.	• Utilize eye protection and leather or heavy rubber (Petroflex or equivalent) gloves. Maintain a safe distance from the discharge of the spray wand. Never point the wand at another person nor use for personal decontamination.

AHA – 09 (continued)

Activity	Potential Hazards	Recommended Controls
	Back Injury from Materials Handling	• Use proper lifting techniques • Loads greater than 50 pounds require assistance or mechanical equipment • Prior to lifting, check the load for jagged or sharp edges • Avoid torso twisting motions while handling or moving loads
	Thermal Stressors (i.e. heat stress, cold stress)	• Employees will have appropriate clothing for variable weather • Use of long sleeves or application of sunscreen with a high sun protection factor (SPF) on exposed skin encouraged • Employees will take breaks and drink plenty of fluids to prevent heat stress • Warming breaks will be permitted as necessary to prevent cold stress
	Inclement weather (Thunderstorms and tornadoes)	• Halt activities immediately and take cover during thunderstorm or tornado warnings, shelter in a building if possible, stay away from windows • If outdoors, stay close to the ground • Listen to radio or television announcements for pending weather information • Do not try to outrun a tornado on foot or in a vehicle
	Biological hazards (insects, spiders etc.)	• Workers will inspect the work area carefully and avoid placing hands and feet into concealed areas Look in direction of travel for biological hazards to avoid
Equipment Used	**Inspection Requirements**	**Training Requirements**
Vehicle and site equipment • Drill rigs • Excavator • Pickup trucks or vans • Canoe or small boat • High temperature pressure washer Hand tools	Inspect all equipment prior to use; refer to manufacturers inspection requirements, if appropriate.	Valid operators license for the class of vehicle. Experience in drill rig and heavy equipment operation to move equipment for decontamination.

Equipment Used	Inspection Requirements	Training Requirements
Photoionization detector	Inspect and calibrate Photoionization detector (PID) daily when in use to monitor intrusive activities.	Experience in operating and calibrating a PID.
Decontamination containment vessels/structures: containers for soil/rock cuttings, groundwater and decontamination residuals from work in source areas	Prior to each use, inspect any decontamination containment vessel or structure. Inspect any container prior before depositing any soil/rock cuttings, groundwater or decontamination residuals from work in source areas.	No specific training required.
		General: • Review of Site-Specific Health and Safety Plan and Activity Hazard Analyses • Hazardous Sites Worker training per 29CFR 1910.120 • Initial site/task specific training. Daily Safety Meetings.

ATTACHMENT C: HEALTH AND SAFETY PLAN PRE-ENRTY BRIEFING ATTENDANCE AND DAILY SAFETY MEETING FORMS

Health and Safety Plan Pre-Entry Briefing Attendance Form

Nike Battery Site PR-58

North Kingstown, RI

Conducted by:		**Date Performed:**	
Topics Discussed:	1. Review of the content of the APP/SSHP/AHA (Required)		
	2.		
	3.		
	4.		

Printed Name	Signature	Time On-site	Time Off-site	Representing

ATTACHMENT D: ACCIDENT INVESTIGATION FORM

STONE ENVIRONMENTAL INC.

Incident/Accident/Injury/Illness Reporting Form

REPORTING SECTION

Form completed by: _____ Date completed: _____

Employee Name: _____ Group: _____ Reported to: _____

Location, Date and Time of Incident or Accident: _____

Description of Events Leading up to Incident or Accident: _____

_____

Equipment/Tool/Vehicle involved in the incident or accident: _____

Was Equipment/Tool/Vehicle defective? _____ If yes, describe: _____

Describe the damage or injury. _____

If necessary, please attach police report or other reporting documentation.

Was hospitalization required? _____ If yes, where? _____

Name and Address of Physician and Hospital: _____

Group Leader's Name: _____ Date of Report: _____

INVESTIGATION SECTION

Could the incident or accident have been prevented? ☐ YES ☐ NO If yes, how? _____

_____

Was Safety Equipment provided? ☐ YES ☐ NO Was it used properly? ☐ YES ☐ NO

Was any work time lost? ☐ YES ☐ NO If yes, how much time? _____

Workers Compensation Insurance Notified? ☐ YES ☐ NO

What actions have been taken to prevent similar incidents or accidents? List corrective actions, recommended changes to procedures/equipment/signs to prevent re-occurrences of this type: _____

_____

_____

Employees Signature: _____ Date: _____

Group Leaders Signature: _____ Date: _____

Safety Committee representative: _____ Date: _____

Other Comments: (if necessary attach additional sheet)

ATTACHMENT E: CURRENT TRAINING CERTIFICATIONS

JOSEPH SCHMIDL, P.G., C.G., C.W.S.

EDUCATION

University of Massachusetts at Lowell
M.S., Environmental Studies, College of Civil Engineering, 1995

University of Massachusetts at Amherst
B.S., Geology, 1982

WORK EXPERIENCE

Stone Environmental, Inc.
Montpelier, Vermont, USA
Senior Project Manager, 08/2012 – Present

Manage environmental programs and large project contracts for clients. Manage multidiscipline project teams and serve as senior technical resource for hydrogeology, site conceptual model development, contaminant fate and transport evaluation, risk assessment, and remedial action design and implementation.

Blackwater Environmental Design
Salisbury, New Hampshire
Designer/Scientist, 04/2003 – Present

Designed environmentally sensitive septic systems, performed soil tests, percolation tests, and septic system inspections. Performed wetland delineations and permitting for residential clients.

Weston Solutions, Inc
Concord, New Hampshire
Technical Manager, 09/1997 – 07/2012

Conducted environmental investigations for federal, state, and local government, and commercial clients under federal and state regulations. Conducted and managed geological, hydrogeological, hydrological, and wetland investigations. Lead sampling activities for groundwater, surface water, sediment, soil, and soil vapor samples, including real-time soil analysis. Developed work plans, coordinated field staff and subcontractors, regularly interacted with clients, interpreted results, reporting, technical review of deliverables, and management of project budgets. Specialized in development of multimedia conceptual site models.

Roy F. Weston, Inc.
Burlington, Massachusetts
Project Leader, 05/1995 – 09/1997

Conducted pre-remedial site assessment projects under EPA Region 1's Superfund Technical Assessment and Response Team (START). Managed six junior staff conducting site assessment projects, including technical review of deliverables, and schedule and budget tracking. Project leader for the Hazard Ranking System packages produced for EPA Region 1. Participated in evaluations of dozens of sites in New England and sites investigated under EPA's Brownfields Program.

Roy F. Weston, Inc.
Wilmington, Massachusetts
Project Geologist, 08/1994 – 05/1995

Conducted pre-remedial site assessment projects under EPA Region 1's Alternative Remedial Contract Strategy (ARCS) program. Projects included numerous site inspections (SIs), including initiation of 12 SIs conducted simultaneously in the Farmington Industrial Park in Connecticut. Supported remedial investigations at the former Pease Air Force Base in Portsmouth, New Hampshire. Duties also included a number of site investigation projects conducted to satisfy state regulations in Connecticut and Massachusetts.

TRC Companies, Inc.
Lowell, Massachusetts
Geoscientist, 07/1991 – 08/1994

Conducted pre-remedial site assessment projects under EPA Region 1's ARCS program. Projects included numerous SIs, RCRA/CERCLA Preliminary Assessments, and two Hazard Ranking System documentation records. Performed technical oversight of responsible party-led projects and a number of site investigation projects under state regulations in Connecticut, Kentucky, and Massachusetts.

NUS Corporation
Bedford, Massachusetts
Geologist, 12/1990 – 07/1991

Conducted pre-remedial site assessment projects under EPA Region 1's dedicated Field Investigation Team (FIT) contract.

Boy Scouts of America,
Dalton and Chicopee, Massachusetts
District Executive, 09/1984 – 12/1990

Served as professional representative to volunteer leaders and chartered organizations. Performed public relations, recruiting and training of volunteer leaders, fundraising, and management of summer camp operations.

ADDITIONAL EDUCATION

Preliminary Assessment and Site Assessment Training, EPA Region I, 1992.

Hazard Ranking System Training, EPA, Kansas City, Kansas, 1993.

Dangerous Goods Shipping, renewed 2010.

Bloodborne Pathogens Training—Initial, OSHA 29 CFR 1910.1030, (1995); refresher, Weston, 2012.

Confined Space Training—Entrant, Attendant, Non-Entry Rescue, OSHA 29 CFR 1910.146, Weston, 1996.

Trenching/Excavation Competent Person Training Course, OSHA 29 CFR 1026 Subpart P, Weston, 2010.

Fall Protection Competent Person Training Course—Initial, OSHA 29 CFR 1926 Subpart M, Weston, 2011.

Asbestos Inspector Title II, TSCA, Institute for Environmental Education, Inc., 2011.

Hazardous Waste Management and Shipping for Environmental Professionals, 40 CFR 265.16, Eduwhere, 2001.

Sample Shipping, Weston, 2009.

Underground Utilities Competent Person Training, Weston, 2010.

ICS-100 Introduction to Incident Command System, FEMA, 2010.

ICS-200 Basic Incident Command System, FEMA, 2010.

Lead Awareness Training, Weston, 2009.

Behavior-Based Safety Training – Phase I, Weston, 2007.

IATA/DOT Shipping Advisor Training—Initial, 2002; refresher, Weston, 2010.

DOT Hazardous Materials Training, 49 CFR 172 Subpart H, HAZMATEAM, Inc., 2008.

First Aid/CPR/AED Training, American Red Cross, 2011.

8-Hour Hazardous Waste Refresher Course, OSHA 29 CFR 1910.120(e)(8), Weston, 2011.

40-Hour Hazardous Waste Site Training Course, OSHA 29 CFR 1910.120(e)(3), NUS Corporation, 1990.

8-Hour Managers and Supervisors Course (SHSC), OSHA 29 CFR 1910.120(e)(4), Weston, 1994.

10-Hour Construction Industry Outreach Training, 29 CFR 1926, Weston, 1998.

Shipping and Transporting Dangerous Goods—Administrative / Field Personnel. Refresher, Weston Solutions, Inc., Manual of Procedures for Shipping and Transporting Dangerous Goods Training Course, 49 CFR 172, Subpart H, Weston, 2011.

Dangerous Goods Shipping Advisors Refresher, 49 CFR 172 Subpart H, HAZMATEAM, 2010.

CERTIFICATIONS

Professional Geologist, State of New Hampshire, License Number 47.

Certified Geologist, State of Maine, Certification Number GE436.

Certified Wetland Scientist, State of New Hampshire, License Number 263.

Licensed Designer of Subsurface Disposal Systems, State of New Hampshire, Number 1547.

Licensed Site Professional, State of Massachusetts, License Number 4442.

OSHA 40-hour certification

OSHA Site Supervisor certification

OSHA Construction Manager certification

Non-Entry Confined Space Entry certification

Asbestos Inspection certification.

SKILLS

Computers: fluent with Microsoft Office (Word, Excel, Powerpoint, Access); CADopia IntelliCAD; ESRI ARCView Geographic Information System; ProUCL and MAROS statistical software.

PROFESSIONAL AND COMMUNITY ACTIVITIES

Chairman of Planning Board, Town of Salisbury, New Hampshire.

PROJECT EXPERIENCE

US Environmental Protection Agency
Walton & Lonsbury Superfund Site, Attleboro, MA: Provided wetlands compliance support.

West Street Property Site, Hatfield, MA: Performed wetlands delineation and prepared a wetlands restoration plan.

Former Chlor-Alkali Facility, Berlin, NH: Lead Geologist for Remedial Investigation/Feasibility Study for this elemental mercury release site.

Eighteen Mile Creek Superfund Site, Lockport, NY: Performed wetlands delineation to support Hazard Ranking System evaluation.

St. Croix Alumina Superfund Site, St. Croix, USVI: Performed wetlands delineation and environmental groundwater, surface water and sediment sampling.

US Army Corps of Engineers
Tom Nevers Formerly Used Defense Site, Nantucket, MA: Prepared Environmental Protection Plan component of Work Plan for UXO survey and removal.

Former LO-58 Site, Caribou, ME: Project Manager for Supplemental Site Investigation involving hydrogeophysical survey and interpretation of bedrock groundwater contamination.

GE/Housatonic River Site, Pittsfield, MA: Technical review of PRP submittals for EPA via USACoE contract. Coordinated activities with Massachusetts Department of Environmental Protection staff, for this multi-jurisdictional site managed under a Consent Decree.

Air Force Center for Engineering and the Environment (AFCEE)
Former Landfill A, Westover Air Reserve Base, Chicopee, MA: Prepared Activity and Use Limitation paperwork and Response Action Outcome Statement for site closure.

Former Construction Rubble Site A, Westover Air Reserve Base, Chicopee, MA: Project Manager for Supplemental Site Investigation involving Field X-Ray Fluorescence survey and Phase II Report.

Explosive Ordinance Disposal Proficiency Range, Westover Air Reserve Base, Chicopee, MA: Project Manager for Supplemental Site Investigation involving subsurface investigation of light, non-aqueous phase liquid, and Phase II Report.

Remedial Investigation/Feasibility Study (RI/FS), Former Chlor-Alkali Facility Site, Berlin, NH, U.S. Environmental Protection Agency (EPA) Region 1, Lead Hydrogeologist

As a Team Subcontractor to Nobis Engineering, Inc., provided technical direction for the investigation of elemental mercury contamination at this former chemical plant site, where elemental mercury discharge via bedrock fractures to the Androscoggin River has occurred. Selected subsurface investigation locations (soil borings and test pits). Reviewed subsurface data collected during monitoring well installation and well development. Directed test pitting investigations, including the test pit excavated in November 2011, during which the disposal location of the former chlor-alklai cells was discovered, along with the elemental mercury they contained. Performed bench-scale testing of hypotheses regarding mercury fate and transport in groundwater. Interpreted groundwater hydrogeological and analytical results, and prepared a summary report for the site. [9-07 to present; WESTON; Proj. No. 12884.013.001 to 003]

Wetland Mapping and Sampling, St. Croix Alumina Site, St. Croix, U.S. Virgin Islands, EPA Region 2, Wetland Scientist

Conducted ground-verification of mapped forested, shrub-scrub, and emergent estustrine wetlands, as well as the confirmation of the location of the downstream surface water pathway in support of a Hazard Ranking System (HRS) documentation record. Wetland extents within the approximately 1-square-mile site were documented in 2 days. Work was accomplished ahead of schedule using only 50% of the estimated level of effort. Following the wetland identification, collected sediment and surface water samples from 30 locations in a 3-day effort [9-11; WESTON; Proj. No. 20405.012.013.1455]

Removal Assessment Support, Former Grand Union Site, Fort Edward, NY, EPA Region 2, Lead Hydrogeologist

Conducted a multimedia subsurface soil and groundwater investigation of a former grocery store property, which may have received fill material from a nearby polychlorinated biphenyl (PCB) facility. Advanced 12 soil borings to depths of 16 feet below ground surface (bgs) and 20 test pits to depths of 8 feet bgs on two potentially impacted properties. Logged soil borings and test pits, and identified buried soil horizons that allowed the extent of fill on the properties to be determined. Also installed two overburden groundwater monitoring wells, and collected soil and groundwater samples to characterize potential risks. Soil samples were analyzed by EPA Region 2's laboratory. Performed an elevation survey for the newly installed wells. Project was completed within budget, and in a

total of 7 working days. [10-11; WESTON; Proj. No. 20401.132.022.5092]

Removal Assessment Support, Jewett White Lead Company Site, Staten Island, NY, EPA Region 2, Project Manager

Conducted a multimedia subsurface soil, sediment, surface-water, and groundwater investigation of a former white lead manufacturing facility. Advanced approximately 25 soil borings and 25 test pits on two impacted properties to depths of 8 feet bgs, installed 5 overburden groundwater monitoring wells, and collected soil, sediment, surface-water, and groundwater samples to characterize potential risks. Soil samples were analyzed on-site for lead utilizing a field portable X-ray fluorescence (FPXRF) unit, with a fraction of the samples submitted to EPA Region 2's laboratory for confirmatory analysis. Performed statistical analysis of the soil lead results to determine usability of FPXRF results. Compiled the results into a Removal Actions Alternatives Report, one each for both properties. Reports included evaluation of up to five remedial options. Project was completed under budget, and within an aggressive 4-month time period. [7-10 to Present; WESTON]

Wetland Delineation and Mapping, 18-Mile Creek Site, Lockport, NY, EPA Region 2, Wetland Scientist

Conducted a delineation of forested and shrub-scrub palustrine wetlands in support of a HRS documentation record. Wetland frontage to approximately 6 stream miles of 18-Mile Creek totaling approximately 1 mile was delineated in a single day according to U.S. Army Corps of Engineers (USACE) Wetlands Delineation Manual Y-87-1 guidelines. Wetland boundary points were located using a Trimble global positioning system (GPS) unit. Work was accomplished ahead of schedule using only 70% of the estimated level of effort. [6-11; WESTON; Proj. No. 20405.012.013]

Removal Action Support, Various Locations in New Jersey and New York, EPA Region 2, Site Manager

Conducted a variety of removal action support tasks for WESTON's Removal Support Team (RST)-2 in Edison, NJ. Activities included removal support (contractor oversight, air monitoring, confirmation sampling) and Administrative Record preparation. [4-11 to 6-11; WESTON; Proj. No. 20401.122]

Wetland Delineation/Restoration Planning, West Street Site, Hatfield, MA, EPA Region 1, Wetland Scientist

Conducted a delineation of forested and shrub-scrub palustrine wetlands in advance of site remedial activities. One area of wetlands totaling approximately 4 acres was delineated in a single day according to USACE Wetlands Delineation Manual Y-87-1 guidelines. Wetland boundary flags were placed for later surveying. Plant species were inventoried to support the development of a Wetland Restoration Plan. [9-09 to Present; WESTON; Proj. No. 20114.056.998]

Disposal Site Closure, Confidential Client Sites, Wilmington, MA, EPA Region 1, Apprentice to Licensed Site Professional (LSP)

Under the authority of a WESTON LSP, conducted a series of investigations at two adjacent disposal sites to satisfy requirements of the Massachusetts Contingency Plan (MCP) (30 CMR 40.0000). The property to be investigated was formerly owned by WESTON's confidential client, which resulted in a complicated project approval process that included the current owner of the property. One disposal site was the site of a release of PCB-containing transformer oil. The other disposal site was an area of contaminated soil and groundwater resulting from contaminated fill beneath a 20-year-old building. Designed and performed or directed a series of iterative soil boring and well installation programs, and soil and groundwater sampling activities. Deliverables included Phase I Initial Site Investigations, Phase II Comprehensive Site Assessments, Phase III Remedial Action Plans, Phase IV Remedy Implementation Plans, and Response Action Outcome Statements to support site closure. In addition, prepared deliverables to satisfy EPA Region 1 Toxic Substances Control Act (TSCA) requirements pursuant to PCBs present at the sites. [2004 to Present; WESTON; Proj. No. 02181.167]

Wetland Delineation, Caryville Mill Superfund Site, Bellingham, MA, EPA Region 1, Wetland Scientist

Conducted a delineation of forested palustrine and riverine wetlands in advance of site remedial activities. Three separate areas of wetlands totaling approximately 40 acres were delineated in a single day according to USACE Wetlands Delineation Manual Y-87-1 guidelines. Wetland boundary flags were located using a sub-meter accurate GPS, and a wetlands boundary map was delivered to the client within 24 hours to expedite the initiation of remedial activities. [7-09 to Present; WESTON; Proj. No. 12884.016]

Brownfields Site Assessment – Underground Storage Tank (UST) Removal, Frank's Texaco Site, Winchester, NH, Southwest Regional Planning Commission (SWRPC), Project Manager

Directed the investigation of potential petroleum groundwater contamination at this former gasoline station site. Prepared generic and

site-specific Quality Assurance Project Plans (QAPPs) for submission to EPA Region 1 Brownfields Section for review. Determined the nature and extent of subsurface soil and groundwater contamination in accordance with New Hampshire Department of Environmental Services (NHDES) regulations (Env-Or 600 and 800). WESTON personnel performed soil sampling, groundwater sampling, and documented groundwater flow direction for the site. In addition, directed the removal of two 6,000-gallon USTs and one 1,000-gallon UST from the site, and performed post-removal confirmatory sampling. Presented hydrogeological and analytical results in a summary report for the site. [10 06 to 6-07; WESTON; Proj. No. 13877.001.002]

RIs, Westover Air Reserve Base (ARB), Chicopee, MA, Air Force Center for Engineering and the Environment (AFCEE), 4P Architect-Engineer (A-E) Contract, Project Manager

Performed environmental investigations of a number of disposal sites under contract to AFCEE pursuant to compliance with the MCP. Activities included review of previous investigations and analytical results; evaluation of existing analytical results for data usability and representativeness in compliance with Massachusetts Department of Environmental Protection (MassDEP) guidance; development of a sampling plan to eliminate data gaps; execution of the sampling program; incorporation of the new analytical results to each site data set; support to subcontracted risk assessors in performing human health and/or environmental risk characterizations; completion of revised MCP deliverables; and interaction with MassDEP personnel in addressing comments on MCP deliverables. In most cases, WESTON started work on these projects after another AFCEE contractor had recommended, and MassDEP had approved, extensive and expensive remedial actions to close these disposal sites under the MCP. By collecting additional environmental samples and eliminating data gaps, WESTON was able to perform much less conservative risk assessments (relying on data rather than conservative assumptions) and document that no remedial activities were required for disposal site closure under the MCP. This approach saved AFCEE 90% of its disposal site closure costs. [5-07 to Present; WESTON; Proj. Nos. 20077.043.093, .150, .155; 20077.048.030]

Hydrogeological Investigation, Maine Formerly Used Defense Sites (FUDSs), Caribou and Perham, ME, USACE, New England District (CENAE), Project Manager

Under contract to CENAE, performed detailed hydrogeological investigations of two FUDSs located in northern Maine with the specific objective of determining the interconnection between impacted bedrock drinking water wells. The investigation included review of previous investigations and analytical results, subcontracting of geophysical and hydrophysical surveys of the impacted drinking water wells, sampling of individual fracture zones based on the results of the geophysical and hydrophysical surveys, and the interpretation of the analytical, geophysical, and hydrophysical data to create a Conceptual Site Model (CSM) for the sites. WESTON interacted with Maine Department of Environmental Protection (MEDEP) personnel as well as CENAE in addressing comments on Work Plans and other deliverables. [3-07 to Present; WESTON; Proj. No. 03886.184.001]

Long-Term Monitoring Program Support, Woodstock, CT, EPA Region 1, Remedial Action Contract (RAC), Project Manager

Provided technical support to EPA Region 1 under its RAC (as a subcontractor to Nobis Engineering, Inc.) at the Linemaster Switch Corporation Superfund site in Woodstock, CT. Support included oversight/technical review of responsible party (RP) contractor field activities and deliverables. WESTON performed oversight of RP contractor field activities during two semiannual monitoring rounds in November 2007 and May 2008. WESTON reviewed three semiannual monitoring reports and one proposal for changes to the long-term monitoring program and provided technical comments to EPA Region 1. The project was completed significantly under budget and within schedule. [9-07 to Present; WESTON; Proj. Nos. 12884.010.001, .003, .008, .010, .011]

Emergency Response Support, Stoddard, NH, EPA Region 1, Superfund Technical Assessment and Response Team (START), Septic System Designer

Provided technical support to EPA Region 1 under its START contract at the Electrosonic Superfund site in Stoddard, NH. Support included the design of a replacement septic system for the facility following removal of the existing system due to contamination with heavy metals. The design was constrained by a small replacement area, bounded by a surface water body. WESTON also provided support in obtaining the required state and local approvals for the septic system design. [3-05 to 9-06; WESTON; Author doesn't have project number]

Hurricane Rita Response Support, Marsh Operations, Chambers County, TX, EPA Region 6 START, Wetland Scientist

Provided technical support to EPA Region 6 under its START contract at various locations in Chambers County, TX. Support included the identification of sensitive wetlands during emergency response operations, and development of alternative access plans in order to reduce environmental impacts by recovery equipment. WESTON scouted ahead of emergency response contractors, and identified routes for heavy equipment that avoided sensitive wetland environments to the greatest extent possible. WESTON then documented pre- and post-removal conditions, as well as performed removal documentation. [10-08 to 11-08; WESTON; Proj. No. 20406.012.030]Hurricane Rita Response Support, Cameron Parish, LA, EPA Region 6 START, Wetland Scientist. Provided technical support to EPA Region 6 under its START contract at various locations in Cameron Parish, LA. Support included the identification of sensitive wetlands during emergency response operations, and development of alternative access plans in order to reduce environmental impacts by recovery equipment. WESTON scouted ahead of emergency response contractors, and identified routes for heavy equipment that avoided sensitive wetland environments to the greatest extent possible. WESTON then documented pre- and post-removal conditions, as well as performed removal documentation. [11-06; WESTON; Author doesn't have project number]

RI Support, Pittsfield, MA, EPA Region 1, Project Manager

Provided technical support to regulatory agencies (EPA and MassDEP) at the General Electric (GE)/Housatonic River site in Pittsfield, MA. Support included technical review of Work Plans submitted to the agencies for compliance with approved Sampling and Analysis Plans, and the Consent Decree (CD) that promulgates the remedial actions at the site. Reviewed Work Plans for soil sampling, subsurface drilling and soil sampling, monitoring well installation, and groundwater sample collection; and provided the comments and recommendations to EPA for inclusion in approval letters. Support also included numerous research activities regarding site background information, with particular emphasis on site hydrogeology. Met regularly with EPA, MassDEP, and other WESTON Project Managers to review progress on the project. Acted as an interface with GE contractors to ensure that work was completed according to approved Work Plans, and helped develop alternatives as site conditions warranted. Provided technical support to CENAE/EPA and MassDEP through technical review of GE deliverables for compliance with the CD and Statement of Work (SOW) for Removal Actions Outside the Housatonic River, from 2001 to the present. Reviewed Removal Action Area (RAA) deliverables, including: Pre-Design Investigation Reports, Conceptual and Final Removal Design/Removal Action Work Plans, and Final Completion Reports; reviewed Groundwater Management Area (GMA) deliverables, including Baseline Groundwater Monitoring Reports, Interim Groundwater Monitoring Reports, Nonaqueous Phase Liquids Monitoring Reports, and Long-Term Groundwater Monitoring Proposals, providing the comments and recommendations to EPA for inclusion in approval letters for the subject deliverables. Support included numerous research activities regarding site background information, with particular emphasis on site hydrogeology and the distribution of nonaqueous phase liquids (NAPLs), including recommendations for supplemental groundwater investigations to address data gaps. Independently plotted groundwater GE analytical results, combining results from adjacent GMAs, to provide additional perspective to EPA regarding contaminant nature and extent. Worked closely with EPA Remedial Project Managers (RPMs) to provide background information for technical meetings. Facilitated meetings among EPA, MassDEP, GE, and their consultants to discuss topics that were incompletely described in the CD and SOW, and resolving differences of opinion. Performed independent spatial averaging calculations for RAAs to not only confirm GE's compliance with the CD, but also to test hypotheses regarding optimal soil removal results. [1998 to Present; WESTON; Proj. No. 20124.001.098]

Environmental Investigation, Former Chlor-Alkali Facility Site, Berlin, NH, NHDES, Project Manager

Directed the investigation of potential elemental mercury groundwater contamination at this former chemical plant site. Investigated the source of elemental mercury discharge via bedrock fractures to the Androscoggin River. Managed drilling subcontractor for monitoring well installation and well development. WESTON personnel performed groundwater sampling and hydraulic conductivity testing for the site. In addition, coordinated efforts with WESTON START personnel who were conducting a concurrent preliminary assessment/site inspection (PA/SI) for EPA Region 1. Interpreted groundwater hydrogeological and

analytical results, and prepared a summary report for the site. Managed three subsequent, annual mercury removals from bedrock fractures along the bank of the Androscoggin River. [6-04 to 4-07; WESTON; Proj. No. 20111.001.001 to .006]

Preparation of HRS Documentation Record, Pownal Tannery Site, Pownal, VT, EPA Region 1 START, Project Leader

Worked at several stages of the HRS evaluation of this site by EPA Region 1. Initially conducted environmental sampling at the site in 1993, and prepared the Site Inspection Prioritization (SIP) of the site under TRC Environmental Corporation's Alternative Remedial Contracting Strategy (ARCS) contract for EPA Region 1. Subsequently prepared a draft HRS Documentation Record for the site, which was shelved at the request of the Vermont Department of Environmental Conservation (VDEC). Both efforts required the review of extensive background data collected by VDEC and EPA Region 1 as part of Resource Conservation and Recovery Act (RCRA), National Pollutant Discharge Elimination System (NPDES), and Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) regulatory activities, including analytical results collected by EPA Region 1 Technical Assistance Team (TAT) during concurrent emergency removals from the site. In 1996, directed technical staff in updating the previous draft HRS Documentation Record at the request of EPA Region 1 under WESTON's START contract. Assisted technical staff with cost tracking and scheduling, reporting monthly to the client regarding deviations from schedule and budget amendments. Duties included technical review of draft and final reports. Provided direction during field activities to ensure compliance with the assigned EPA SOW and EPA guidance documents, and the collection of HRS-quality field data. START delivered the package on time and under budget. [1996 to 1997; WESTON; Author doesn't have project number]

Environmental Investigation, Former Greene Tannery Site, Milton Mills, NH, NHDES, Project Manager

Directed the investigation of potential remnant groundwater contamination at this former tannery site, following remedial activities, in consideration of possible redevelopment. Obtained drilling subcontractor for monitoring well installation, and performed well development and groundwater sampling for the site. Required the review of extensive background data collected by NHDES and EPA Region 1 as part of RCRA, NPDES, and CERCLA regulatory activities, including analytical results collected by EPA Region 1 TAT during previous emergency removals from the site. In addition, obtained environmental data from WESTON START personnel, who were conducting a concurrent SIP for EPA Region 1. Interpreted groundwater analytical results, as well as WESTON START soil, sediment, groundwater, and surface water analytical results (prior to the SIP submission to EPA Region 1), and prepared a summary report for the site. [1997 to 1998; WESTON; Author doesn't have project number]

Preliminary Assessment-Plus Inspections, Multiple Commercial and Industrial Properties, New England, Site Leader

Conducted facility inspections to ensure compliance with EPA Region 1 regulations under CERCLA and RCRA, using National Corrective Action Prioritization System (NCAPS) criteria. Performed facility audits of hazardous materials (waste handling, storage, and disposal). Included inspection of hazardous materials storage areas, process areas, and waste accumulation or disposal areas at each facility. [1992 to 1993; TRC]

RI/Comprehensive Site Assessment Oversight, EPA Region 1, Field Team Leader

Coordinated the technical aspect of providing oversight of drilling and monitoring well installation for EPA Region 1 at its highest priority site from 1998 to the present. Developed an electronic report format to keep EPA, MEDEP, USACE, New England District (CENAE), and WESTON Project Managers abreast of daily progress on the project. Acted as an interface with potentially responsible party (PRP) contractors to ensure that work was completed according to approved Work Plans, and helped them develop alternatives as site conditions warranted. Also provided EPA Region 1 with daily summary of nonaqueous liquids noted, soil headspace screening results, and collected split samples to confirm PRP contractor laboratory results. Also conducted technical review of the reports documenting the work that was overseen, correcting errors, and reinterpreting the data presented. [1998 to 2003; WESTON; Proj. No. 20124.001.098]

Groundwater Extraction System Installation, Groveland Wells Superfund Site, Groveland, MA, Metcalf & Eddy/EPA, Project Geologist

Directed subcontractors in advancing pilot borings, installing and developing monitoring wells, and installing and developing five extraction wells (completed in overburden and/or bedrock). Assisted Project Manager with cost tracking and scheduling, and reporting costs to the client. Also assisted with the carbon filtration of wastewater

generated during the field activities, completing the project 1 week earlier than budgeted, and significantly reducing subcontractor costs. [1998; WESTON; Author doesn't have project number]

Site Assessments, New England, EPA Region 1 START, Project Leader

Managed the activities of six technical staff in conducting PAs, SIs, Mini-SIs, and HRS Documentation Records. Assisted technical staff with cost tracking and scheduling, reporting monthly to the client regarding deviations from schedule and budget amendments. Duties included technical review of deliverables such as Task Work Plans/Non-Sampling Recommendation Memoranda, trip reports, and draft and final reports. Provided direction during field activities (including site reconnaissance and facility inspections, and field sampling) to ensure compliance with the assigned EPA SOW. [1995 to 1997; WESTON; Various Project Nos.]

Preparation of HRS Documentation Record, Housatonic River, Pittsfield, MA, EPA Region 1 START, Project Leader

Managed the activities of six technical staff in preparing the HRS Documentation Record for this site. The project was EPA Region 1's highest priority from November 1996 to August 1997. Assisted technical staff with cost tracking and scheduling, reporting monthly to the client regarding deviations from schedule and budget amendments. Duties included technical review of draft and final reports. Provided direction during field activities to ensure compliance with the assigned EPA SOW and EPA guidance documents, and the collection of HRS-quality field data. As deadlines neared, anticipated workload and requisitioned additional staff to meet deadlines for deliverables. EPA Region 1 requested that START complete the project in less than the typical schedule to meet EPA Headquarters proposal deadlines. Further, the site was complicated by the overlapping CERCLA and RCRA programs, both of which had jurisdiction over the GE facility. This led to tasks and comments not typically required for an HRS Documentation Record. Despite these obstacles, START delivered the package on time in every case, receiving written congratulations from the EPA Site Assignment Manager for WESTON's efforts. [1995 to 1997; WESTON; Author doesn't have project number]

Geoprobe® Operation, Various Hazardous Waste Sites in New England, Multiple Clients, Operator

Following manufacturer's guidelines, operated the EPA Region 1 Geoprobe® hydraulic soil sampling unit at various hazardous waste site investigations in New England. Used the unit to collect deep soil samples, groundwater samples, install miniature monitoring wells, and perform continuous soil screening/logging. Performed routine maintenance on the unit, and trained geotechnical staff in its safe operation. [1992 to 1997; WESTON; Proj. Nos. various ARCS and START assignments]

Technical Skills Training, Various Locations, WESTON, Trainer

Performed training in the following areas: EPA's HRS risk assessment model, WESTON's Dangerous Goods Shipping policy supporting International Air Transport Association (IATA) shipping regulations, and operation of the Geoprobe® hydraulic soil sampling unit. Tasks included development of the training curriculum, preparation of training materials, presenting the training course, and follow-up paperwork, as required. [1998 to Present; WESTON; Proj. No. 00834.003]

HRS Documentation Record, South Weymouth Naval Air Station (NAS), MA, EPA, ARCS Contract, Task Manager

Compiled file information to document the presence of hazardous substances. Determined that analytical results of samples collected from the site by third parties did not meet EPA quality assurance/quality control (QA/QC) objectives. Employed an innovative approach to evaluate the site to develop the HRS Documentation Record. The record withstood public scrutiny, and the site was added to the National Priorities List (NPL). [1993 to 1994; TRC]

SIPs, Various Locations, EPA, ARCS Contract, Deputy Project Manager

Documented the occurrence of hazardous materials at CERCLA sites based on file information and on-site reconnaissance and sampling. Provided guidance to Task Managers in preparing sampling plans designed to meet EPA's HRS objectives. Assisted Task Managers to keep projects on schedule and within budget. Performed technical review of project deliverables. [1991 to 1994; TRC]

Site Inspection, Active Manufacturing Facility, Connecticut, EPA, ARCS Contract, Task Manager

Documented the occurrence of hazardous materials (metals and chlorinated solvents) at this Superfund site based on file information, facility inspection, and waste sample analytical results. Obtained access to four adjacent properties to collect groundwater, surface-water, and sediment samples

to document a release from on-site sources. Samples were collected to meet Level IV data quality objectives (DQOs). [1992; TRC]

RI, Drum Disposal Area, Keysburg, KY, Confidential Client, Field Geologist

Directed the installation of soil borings and monitoring wells in the vicinity of a former drum disposal area. Performed geological reconnaissance over a 20 square-mile area downgradient of the site to map the occurrence of disappearing/appearing streams, sinkholes, and other karst features. Collected groundwater samples on-site, drinking-water samples from wells located downgradient of the site, and surface-water samples that documented the release of hazardous substances (chlorinated solvents) to groundwater. [1993; TRC]

Remedial Action, Railroad Yard, Hermon, ME, Bangor-Aroostook Railroad, Field Geologist

Performed oversight of excavation of waste-oil-contaminated sediments from a wetland adjacent to a railroad yard. Performed continuous air monitoring for all personnel on the project. Collected confirmatory soil samples for the excavation to document achievement of remedial goals. Assisted with the construction of a 10 acre landfarm for on-site remediation of petroleum-contaminated sediments. [1992 to 1993; TRC]

Flood Control Study, Saugus River Estuary, Saugus, MA, USACE, Field Geologist

Project was a flood control study to determine sedimentation rates within the estuarine system. Assisted with the identification of appropriate sample locations, based on vegetation type. Collaborated on the design of sampling apparatus and establishment of sampling stations. Collected sediment cores for Cs137 dating analysis, storm event sediment and water samples, and measured river flow rates during tidal sources. [1992; TRC]

Site Assessments, Commercial and Industrial Properties, Connecticut, Confidential Mortgage Firm, Field Geologist

Assisted with environmental assessments at four separate properties in 4 days. Utilized magnetometer and electromagnetic survey equipment to document the location of underground tanks and utilities to facilitate site clearance for subsequent groundwater well installation. Collected groundwater samples from on-site monitoring wells. Prepared results for inclusion in reports within 2 day turnaround schedule. [1993; TRC]

CERTIFICATE OF ACHIEVEMENT

This is to certify that

Joseph Schmidl

Has achieved recognition for completing
OSHA 8hr Annual Refresher Training for 40hr Health & Safety Class
Hazardous Waste Operations and Emergency Response (29 CFR 1910.120)
and Annual Refresher in Ethics Training.

Montpelier, Vermont on 1/7/2013

Signature : Kim B. Watson Date: 1/7/2013

STONE ENVIRONMENTAL INC

American Red Cross

This recognizes that

JOSEPH SCHMIDL

has completed the requirements for

Adult FA/CPR/AED

conducted by

American Red Cross

Date completed: **01/07/2013**

Valid for **2** year(s)

Certificate of Training

This Document Certifies That

JOSEPH SCHMID

Has Successfully Completed The OSHA Required 8 Hour

HEALTH AND SAFETY TRAINING

Complying with OSHA STANDARD 29CFR 1910.120

MANAGER SUPERVISOR

Patrick J. Thornton

Course Director

EPT inc.
Environmental Protection Training, inc.

OCTOBER 25, 1991

Date of Completion

002278

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CERTIFICATE OF ACHIEVEMENT

This is to certify that

Chris Aldrich

Has achieved recognition for completing
OSHA 8hr Annual Refresher Training for 40hr Health & Safety Class
Hazardous Waste Operations and Emergency Response (29 CFR 1910.120)
and Annual Refresher in Ethics Training.

Montpelier, Vermont on 1/7/2013

Signature : Kim B. Watson Date: 1/7/2013

STONE ENVIRONMENTAL INC

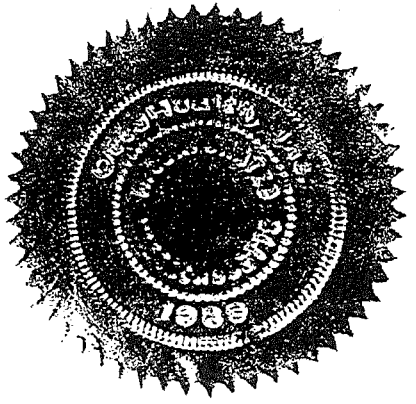
Certificate of Achievement
Presented
To

Christopher D. Aldrich

For the successful completion of a 40 - hour training program
in health and safety, which meets OSHA requirements under
29 CFR 1910.120 for hazardous waste operations.
Course Given By OccuHealth, Inc., Mansfield, Mass. (508) 339-9119

Thomas E. Hamilton

Thomas E. Hamilton, CIH
Course Director, President



April 1990

LEE ROSBERG

EDUCATION

St. Lawrence University, Canton, New York
B.S., Geology, 2005

WORK EXPERIENCE

Stone Environmental, Inc.

Montpelier, Vermont, USA
Project Scientist, February 2007 - Present

Provides assistance for projects as needed. Responsibilities include: acquisition of data and development of technical specifications for application design, report writing and generation, in-house and field technical support, literature, phone, and internet research, lead field work efforts, equipment procurement and maintenance, report/data quality control and field data handling.

CORE$^{DFN\ TM}$ Service Leader II responsibilities include: procurement and maintenance of rock coring equipment, preparing equipment for field work, training personnel, act as field team leader for rock core projects, and assist project manager as needed.

Putney Student Travel

Putney, Vermont, USA
Tanzania Community Service Trip Co-Leader, June 2005 – July 2006

Lead High School students in community service projects during a one month program in Tanzania. Other responsibilities include organizing side trips, guest presentations, maintain work and living supplies, and facilitate group discussions.

G.W. Tatro Construction

Jeffersonville, Vermont, USA
Laborer, May 2004 - October 2006

Laborer on earthwork projects.

ADDITIONAL EDUCATION

Environmental Site Assessments for Commercial Real Estate. ASTM International. 1.4 CEU's, April 26-27, 2012

The Groundwater Pollution and Hydrology Course. Princeton Groundwater, Inc. 3.8 CEU's, March 2-6, 2009

40-Hour OSHA Hazardous Waste Operations and Emergency Response Health and Safety Training, March 1, 2007, 8 hour refresher March 27, 2008.

Study Abroad Semester, Kenya Semester Program, Spring 2004.

CERTIFICATIONS

First Aid and CPR.

HONORS AND AWARDS

Eagle Scout, awarded 1998.

Frederick K. Kilian Scholarship Fund, 2004-2005. Supports students with financial need.

SKILLS

Microsoft Office (Excel, Word, Access, PowerPoint, and Office), Grapher, MODFLOW, ArcView, Adobe Illustrator

Proficient in Swahili.

PROFESSIONAL AND COMMUNITY ACTIVITIES

Member, American Association of Petroleum Geologists, 2005

Member, St. Lawrence University Alpha Tau Omega National Fraternity, 2002 - 2005

St. Lawrence University Geology Club, 2001 – 2005. Vice President Spring 2005. Organization of geology fieldtrips, fundraising, and other events

Kenya Semester Program Applicant Review Committee, Fall 2004. Evaluate students' applications and conduct interviews for acceptance into study abroad program.

PROJECT EXPERIENCE

Contaminated soils excavation at former Kestler's Market, Alburgh, Vermont (2010-2011)
Field Technician for petroleum-contaminated soil removal. Operated excavator, restored Site, and conducted subsequent groundwater sampling.

Lead remedial implementation at former Rhoades Salvage, Milton, Vermont (2012)
Field technician for former salvage yard Site. Field analyzed soil samples to delineate extent of lead contaminated soils. Prepared report on findings. Subsequently operated skid steer to remove contaminated soils.

Soil excavation and disposal at a residence, Randolph, Vermont (2012)
Provided oversight of subcontractor excavating petroleum contaminated soils at a residence. Responsible for field screening of excavation with PID to ensure all impacted soils were removed. Prepared report to VT DEC following completion of field work.

Voluntary Cleanup Site, NY,
Field Geologist for deep Waterloo[APS] and on-site laboratory project. Sampling to depths of 580 feet in sand and gravel aquifer.

Voluntary Cleanup Site, Manchester, UK.
Field Geologist for Waterloo[APS] project. Completed approximately 18 profiles in ten days of depths ranging from 35 to 75 ft bgs.

Vermont Air National Guard Site, Vermont
Field Geologist for SVOC and heavy metals site. Redeveloped and sampled six monitoring wells. Deployed and collected passive diffusion bag samples. Collected surface water and soil samples. Drafted report of analytical results and updated site CSM.

Brownfields Site, Vermont
Field Geologist for PCB, Dioxin, and PAH site. Described and sampled shallow soil cores at over 70 locations in three days. Cached each location with a handheld GPS unit.

ITT Production Facility, New York
Hydrogeologist for CORE[DFN TM] project. Sampled and processed rock core, to be analyzed for chlorinated solvents and physical properties (bulk density, % moisture, TOC, and porosity), at 3 locations in a dolostone formation.

Boeing Research Facility, California
Hydrogeologist for CORE[DFN TM] project. Sampled and processed rock core, at four locations at depths up to 1,400 ft bgs in sandstone and shale formations, to be analyzed for chlorinated solvents, physical properties (bulk density, % moisture, TOC, and porosity), geochemical properties, chloride, and microbial analysis.

Raytheon Production Facility, Massachusetts
Field Geologist for Triad site investigation. Completed dozens of ground water profiles, using Waterloo[APS] technology, during three mobilizations of depths from 60 to >100 ft bgs. Collected as many as 14 groundwater samples in one day.

Honeywell Chemical Manufacturing Plant, Germany
Field Geologist for Waterloo[APS] project. Completed 16 groundwater profiles in three weeks, depths of the profiles ranged approximately between 30 and 55 ft bgs, samples were collected on average every 5 feet.

Ashland Manufacturing Facility, South Carolina
Hydrogeologist for CORE[DFN TM] project. Sampled and processed samples, to be analyzed for VOCs and physical properties (bulk density, % moisture, TOC, and porosity), from unconsolidated deposits, saprolite, and biotite gneiss at eight locations during a week and a half field effort.

Lockheed-Martin Facility, Pennsylvania
Project Manager for CORE[DFN TM] project. Main point of client contact for five week long rock coring and sampling field program. Drilling was conducted within four coreholes, over 400 samples collected for VOC analysis and over 30 collected for physical properties analysis. Responsible for data management and report preparation.

03/01/2007 15:21 1.888.877.7196 Natl Env Trainers Page 1/1

THE NATIONAL

ENVIRONMENTAL TRAINERS

certify that

Lee Rosberg

has satisfactorily passed an exam and completed a 40 hour training course entitled

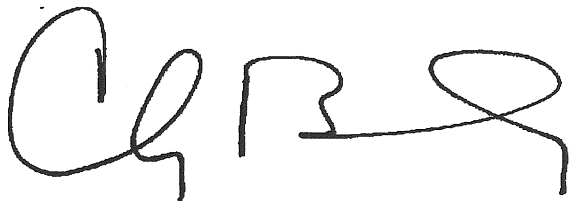
"Hazardous Waste Operations and Emergency Response"

meeting the requirements identified in Title 29 CFR 1910.120 (OSHA HAZWOPER Regulations). This course has been awarded 5.0 Industrial Hygiene CM Points by the American Board of Industrial Hygiene-Approval Number 13334. This course is eligible for 3.33 Continuance of Certification (COC) points from the Board of Certified Safety Professionals.

NATIONAL ENVIRONMENTAL TRAINERS, Inc.

March 1, 2007

Signature of Instructor



Clay A. Bednarz, MS, RPIH

CERTIFICATE OF ACHIEVEMENT

This is to certify that

Lee Rosberg

Has achieved recognition for completing
OSHA 8hr Annual Refresher Training for 40hr Health & Safety Class
Hazardous Waste Operations and Emergency Response (29 CFR 1910.120)
and Annual Refresher in Ethics Training.

Montpelier, Vermont on 1/12/2012

Signature : Kim B. Watson Date: 1/12/2012

STONE ENVIRONMENTAL INC

American Red Cross

This recognizes that

Lee Rosberg

has completed the requirements for

Adult FA/CPR/AED

conducted by

American Red Cross

Date completed: **01/07/2013**

Valid for **2** year(s)

redcross.org

e Instructor's Signature

Alejandro Ibarra

Chapter

Northern Vermont Region

Holder's Signature

Lee Rosberg

S70211

Stock No. 356798

New England Occupational Trainers, Inc.

128 Howland Road - Assonet, MA 02702 - 508-644-5004

Certificate of Completion

this is to certify

Michael Joesph Jordan

has successfully completed the

40 Hour OSHA HAZWOPER Training Course

29 CFR 1910.120

on this 2nd day of February, 1996 at Wellesley, Massachusetts

Certificate No. 0296-0313

Robert Greenlee
Director of Training

Jason Grunke
Vice President, Operations

CERTIFICATE OF ACHIEVEMENT

This is to certify that

Michael Jordan

Has achieved recognition for completing
OSHA 8hr Annual Refresher Training for 40hr Health & Safety Class
Hazardous Waste Operations and Emergency Response (29 CFR 1910.120)
and Annual Refresher in Ethics Training.

Montpelier, Vermont on 11/21/2011

Signature : Kim B. Watson Date: 11/30/2011

STONE ENVIRONMENTAL INC

CONCENTRA Medical Centers

MEDICAL EXAMINER'S CERTIFICATE

I certify that I have examined Michael J Jordan in accordance with FMCSR 49 CFR 391.41-391.49 and with knowledge of the driving duties, I find this person is qualified; and, if applicable, only when:

- ☐ Wearing Corrective Lenses
- ☐ Wearing Hearing Aid
- ☐ Accompanied by a _____ waiver/exemption
- ☐ Driving within an exempt intracity zone (49 CFR 391.62)
- ☐ Accompanied by a Skill Performance Evaluation Certificate
- ☐ Qualified by operation of 49 CFR 391.64

The information I have provided regarding this physical examination is true and complete. A complete exam form with any attachment embodies my findings completely and correctly, and is on file in my office.

SIGNATURE OF MEDICAL EXAMINER	TELEPHONE NO. 802 223-7499	DATE 1/18/12
MEDICAL EXAMINER'S NAME (print) Rebecca Scholl	☐ MD ☐ DO	
MEDICAL EXAMINER'S LICENSE OR CERTIFICATE NO. / ISSUING STATE 055 0030772 / VT	☐ Chiropractor ☐ Advanced Practice Nurse ☐ Physician's Assistant	
SIGNATURE OF DRIVER	DRIVER'S LICENSE NO. 92136213	STATE VT
DRIVER'S ADDRESS (Street, City, State, Zip Code) 548 Big Basin Rd Fayston VT	MED. CERT. EXPIRATION DATE 01/18/2014	

NOTE: Driver MUST carry a copy of this certificate when operating a commercial motor vehicle in accordance with 49 CFR 391.41 (a)

OSCLO

CMCDOTCARD

CONCENTRA Medical Centers

MEDICAL EXAMINER'S CERTIFICATE

I certify that I have examined DAVID J LINARI in accordance with FMCSR 49 CFR 391.41-391.49 and with knowledge of the driving duties, I find this person is qualified; and, if applicable, only when:

- [X] Wearing Corrective Lenses
- [ ] Wearing Hearing Aid
- [ ] Accompanied by a _____ waiver/exemption
- [ ] Driving within an exempt intracity zone (49 CFR 391.62)
- [ ] Accompanied by a Skill Performance Evaluation Certificate
- [ ] Qualified by operation of 49 CFR 391.64

The information I have provided regarding this physical examination is true and complete. A complete exam form with any attachment embodies my findings completely and correctly, and is on file in my office.

SIGNATURE OF MEDICAL EXAMINER: Rebecca A. Scholl PA-C	TELEPHONE NO.: 802-223-7499	DATE: 3/18/11
MEDICAL EXAMINER'S NAME (print): Rebecca Scholl	[ ] MD [ ] DO	
MEDICAL EXAMINER'S LICENSE OR CERTIFICATE NO. / ISSUING STATE: 055-0030772 VT	[ ] Chiropractor [ ] Advanced Practice Nurse [X] Physician's Assistant	
SIGNATURE OF DRIVER	DRIVER'S LICENSE NO.: 22240960	STATE: VT
DRIVER'S ADDRESS (Street, City, State, Zip Code): 1307 JUNCTION RD BERLIN VT 0560	MED. CERT. EXPIRATION DATE: 03/18/2013	

NOTE: Driver MUST carry a copy of this certificate when operating a commercial motor vehicle in accordance with 49 CFR 391.41 (a)

CMCDOTCARD

American Red Cross

Together, we can save a life

This recognizes that

NAME MICHAEL JORDAN

has completed the requirements for

FIRST AID

conducted by

JOHNSON COMPANY

Date completed FEB 34, 2008

The American Red Cross recognizes this certificate as valid for 3 year(s) from completion date.

American Red Cross

Together, we can save a life

This recognizes that

NAME MICHAEL JORDAN

has completed the requirements for

ADULT CPR/AED

conducted by

JOHNSON COMPANY

Date completed FEB 14, 2008

The American Red Cross recognizes this certificate as valid for 1 year(s) from completion date.

CERTIFICATE OF ACHIEVEMENT

EBI TRAINING SERVICES CAMBRIDGE, MA

MANAGEMENT/SUPERVISOR COURSE

THIS IS TO CERTIFY THAT

Michael Jordan

HAS SUCCESSFULLY COMPLETED 8 HOURS OF MANAGEMENT/SUPERVISOR TRAINING IN HAZARDOUS WASTE OPERATIONS AND EMERGENCY RESPONSE IN ACCORDANCE WITH THE OCCUPATIONAL SAFETY AND HEALTH ADMINISTRATION REGULATIONS 29 CFR 1910.120 AND 1926.65

Julie MacDonald

Training Coordinator, Training Services

September 14, 1998

THE NATIONAL ENVIRONMENTAL TRAINERS

certify that

Willapa Waterstrat

has satisfactorily passed an exam and completed a 40 hour training course entitled
Hazardous Waste Operations and Emergency Response
meeting the requirements identified in Title 29 CFR 1910.120 (OSHA HAZWOPER Regulations). This course has been awarded 5.0 Industrial Hygiene CM Points by the American Board of Industrial Hygiene-Approval Number 13334. This course is eligible for 3.33 Continuance of Certification (COC) points from the Board of Certified Safety Professionals.

NATIONAL ENVIRONMENTAL TRAINERS, Inc.

October 2, 2007

Course Number 1003, Awarded 40 PDH's
Florida Board of Professional Engineers CEU Provider Number 0004284

www.nationalenvironmentaltrainers.com

Signature of Instructor

Clay A. Bednarz, MS, RPIH

CERTIFICATE OF ACHIEVEMENT

This is to certify that

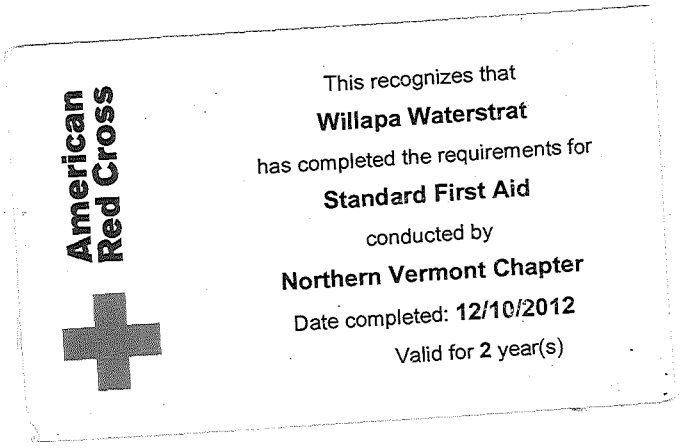
Will Waterstrat

Has achieved recognition for completing
OSHA 8hr Annual Refresher Training for 40hr Health & Safety Class
Hazardous Waste Operations and Emergency Response (29 CFR 1910.120)
and Annual Refresher in Ethics Training.

Montpelier, Vermont on 12/10/2012

Signature : Kim B. Watson Date: 12/10/12

STONE ENVIRONMENTAL INC



American Red Cross

This recognizes that

Willapa Waterstrat

has completed the requirements for

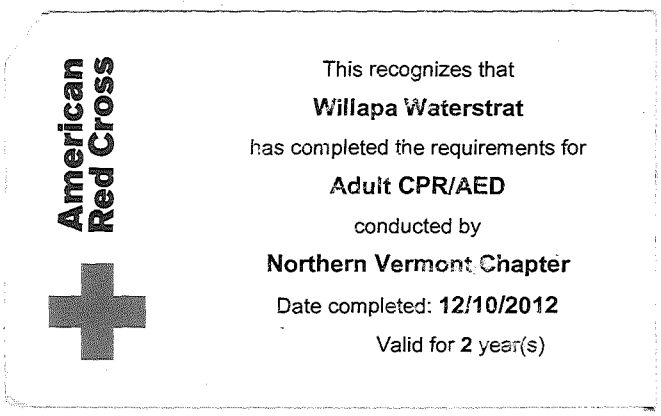
Standard First Aid

conducted by

Northern Vermont Chapter

Date completed: **12/10/2012**

Valid for **2** year(s)



American Red Cross

This recognizes that

Willapa Waterstrat

has completed the requirements for

Adult CPR/AED

conducted by

Northern Vermont Chapter

Date completed: **12/10/2012**

Valid for **2** year(s)

KIM BRYANT WATSON, RQAP-GLP

EDUCATION

Norwich University, Northfield, Vermont
B.S., Environmental Engineering Technology, *cum laude,* 1981
"Environmental Applications of Gas Chromatographic Mass Spectrometry", Indiana University, July 1995
Registered Quality Assurance Professional in Good Laboratory Practices, *RQAP-GLP*, April 2004-2013
National Alliance of Independent Crop Consultants – Provisional Member 2012

WORK EXPERIENCE

Stone Environmental, Inc.
Montpelier, Vermont, USA
Quality Assurance Manager, *Health and Safety Officer* - 10/01 – Present

Manages compliance with Good Laboratory Practices (GLP) and other Quality Assurance (QA) programs such as OECD GLPs. Manages and tracks regulatory requirements associated with The National Environmental Accreditation Program (NELAP), National Environmental Field Activities Program (NEFAP) under TNI (The NELAC Institute), and ISO 17025 Quality Standards. Supervises Stone's QA/QC internal procedures, and performs both internal and external audits.

Acts as the Quality Assurance Unit (QAU) for EPA related environmental and agrochemical studies. Responsible for reviewing outgoing protocols/reports for GLP compliance.

Responsible for providing assistance in writing and reviewing Standard Operating Procedures (SOPs).

Manages corporate quality practices and developed the Quality Management Plan (QMP). Quality Systems Manual (QSM) for Sampling and Analysis

Responsible for developing and implementing quality systems for company processes both project and non-project related. Approves Site Specific and Generic Quality Assurance Project Plans (QAPPs).

Administer the Corporate Health and Safety Management Program, Act as a resource for employees of the company and manage the Safety Committee. Review all accident and injury reports and report to Safety Committee. Review and approve project-specific site Health and Safety Plans for field investigations.

Assists in conducting all aspects of pesticide fate and transport studies under FIFRA groundwater monitoring studies including database management and report writing. She has experience in performing modeling scenarios in CXTFIT2, SETBACK, PRZM3, PE5 and EXPRESS.

Project Management in QA Services with master service agreements with Phoenix Chemistry Services and other consultants which includes all Tiers of data validation, data assessment, data production and auditing (laboratory audits, facility audits). Responsible for the marketing and sales of Stone's QA services such as data validation and outside auditing.

Quality Assurance Officer for the mobile laboratory operations and supports investigations for soil, groundwater and surface water studies, database management, and report writing.

Severn Trent Laboratories
Colchester, Vermont, USA
Quality Assurance Manager, 6/97 – 10/01

Responsible for the overall laboratory quality assurance. Responsible for the continuous development, documentation of QA procedures dealing with the day to day operation of the laboratory, and the implementation of the QA Program for inorganic and organic analyses of environmental samples. In addition, she oversees performance evaluations, certifications, reviewing analytical reports, and following up on corrective action reports. Controlled and reviewed the laboratories' Quality Systems Manual and SOPs.

Trillium, Inc.
Montpelier, Vermont, USA
Quality Assessment Manager, 4/95 – 6/97

Reviewed and validated CLP-type data packages generated in support of sampling analysis program at clients' industrial/commercial plant sites.

Validation was conducted in accordance with EPA's National Functional Guidelines, regional guidelines, other agency procedures, method specific standard operating procedures, and professional judgment as appropriate.

Responsibilities included compilation and evaluation of site data from various on-site analytical programs. Gained experience in design and development of an on-site analytical laboratory. Ensured data was compliant for publication and possible litigation. Prepared and designed project documents such as analytical SOPs and QAPP, and designed and evaluated performance of sampling and analysis projects.

Upon request from USEPA Region I, provided extensive review and comments on Parts I and II of the Region I, EPA-New England Data Validation Functional Guidelines for Evaluation Environmental Analyses.

Facilitated education seminars on environmental chemistry for Engineers and Lawyers.

Inchcape Testing Services, Aquatec Laboratories
Colchester, Vermont, USA
GC/MS Data Review Group Leader, Chemistry Project Director, 6/98 –4/95
QA/QC Data Review Specialist, 11/83 – 6/98

Responsible for a group of seven people working on the review of data, publication, and mass spectral interpretation. Assisted in overseeing daily laboratory operation such as sample handling, method development, analysis, data processing, review and report generation. Analytical experience in the VOA HP5971A MSD GC/MS Systems.

Project Director for USEPA Special Analytical Service Solicitations Contracts, CLP government contracts, and other private client contracts.

LCIC Habitability Study; reviewed final data deliverables and responsible for daily electronic upload of GC/MS analytical data to project bulletin board.

PCB Study, New Bedford, MA: Performed review and quality control of GC/MS analysis for the development of analytical procedures published in "Application of a Mixed-Method Analytical Scheme for Analysis of PCB in Water and Sediment Samples from a Polluted Estuary," Richard A. McGrath, William Steinhauer and Siegfried Stockinger (1987).

Aquatec Laboratories
Extraction Lab Technician, 11/83 – 11/84

Vermont Agency of Environmental Conservation, Solid Waste Program
Montpelier, Vermont, USA
*Air and Solid Waste Technicia*n, 1981 – 1982

Responsible for the environmental engineering design of solid waste and waste disposal facilities and water quality monitoring at the solid waste facilities throughout the State of Vermont.

CERTIFICATIONS/REGISTRATIONS

Registered Quality Assurance Professional in Good Laboratory Practices-RQAP-GLP, 2004-2013

Current Trainer of 40-Hour (29 CFR 1910.120) OSHA Health and Safety Training for hazardous waste operations and emergency response, November, 1995.

ADDITIONAL EDUCATION

2011- TNI FSMO Standard Training (AB Evaluator Training) - February 14, 2011

2011-2010 SQA 27th 26th Annual Meetings

2006 - Uniform Federal Policy for Quality Assurance Project Plans Course #06042

X-MET 3000 Training Program Operation and Radiation Safety, September 2004

GLP Training, Quality System Consultants, Inc. Patricia Royal, M.S.;D.A.B.T., QAP/GLP Registered, January 2002

Gearing Up for NELAP, National Laboratory Training Network, November 1998

HONORS AND AWARDS

Engineering Technology Award, Norwich University, 1981

Employee of the Month – April 19, 2006 and September 2012.

SKILLS

Data validation by various EPA Region's including national guidelines of volatile (GC/MS and GC), semivolatile, inorganic, pesticide, herbicide, polynuclear aromatic hydrocarbons, total petroleum hydrocarbons, alkyltin, and classical chemistry data (soil, water, waste, and air samples) pursuant to EPA's National Function Guidelines, and regional guidelines.

Experience in performing analysis on both GC/MS and GC instrumentation. Field potable X-Ray Fluorescence Spectrum Analyzers.

Experience in performing laboratory audits and preparation and review of QAPP, Federal UFP QAPP and Sampling and Analysis Plans (SAP). Experience in design and development of an on-site analytical laboratory program. Experience in developing eQAPP using ADR.net software and development of SEDD EDDs.

All aspects and sample collection including soil, water and air.

Department of Justice, Expert Witness in environmental chemistry principals.

PROFESSIONAL ACTIVITIES

NAICC - 2012
NEFAB-National Environmental Field Accreditation Executive Committee Member – 2009-present
RQAP-GLP
American Chemical Society
Norwich University Engineering Society
American Society for Quality
Society of Quality Assurance
President and Co-Founder of Chi Beta Chapter of Tau Alpha Phi

PROJECT EXPERIENCE

Design, implementation and maintenance of an on-site analytical program for a reputable engineering firm. Results compiled into a database for statistical analysis.

Validation of analytical data for samples analyzed pursuant to the U.S. EPA Contract Laboratory Program Statement of Work, SW-846, and various other EPA methodologies. Familiar with U.S. EPA functional guidelines for data validation, as well as various regional functional guidelines.

Conducted on-site internal audits of the analytical laboratories. Audits include review of organization and personnel, sample receipt and storage area, general laboratory facilities; analytical instrumentation, data handling, review and documentation; quality assurance program plan and standard operating procedures; and prior performance evaluation data. Co-author of Analytical Laboratory Standard Operating Procedures and the laboratory QSM.

TNI FSMO Standard Training (AB Evaluator Training): February 14, 2011. Two years of experience implementing an accreditation program. Two years' experience developing or participating at a managerial level in FSMO or related accreditation programs. Perform on-site evaluation of an Accreditation Body (AB).

Site investigations performed at specific landfill operations in the State of Vermont. Site investigations included collection of soil from backhoe pits, installation and sampling of groundwater monitoring well, and installation and sampling of groundwater and surface water monitoring points. Asbestos Air monitoring during an asbestos disposal operation.

PRESENTATIONS

Watson, Kim. 2002. GOT ETHICS, Ethics Training, Fraud Prevention and Detection. Presented to NERCSQA November 2002.

Watson, Kim. 2003. Manual Integration Basics, Techniques in Manual Integration. Presented to NY&PA AAEL August 2003

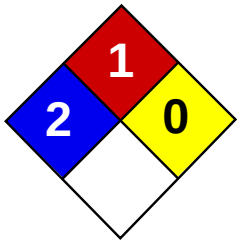
Watson, Kim, 2004. Quality Assurance Training. Data Integrity Procedures and Ethics and Laboratory Quality Assurance. Full Day Workshop for ITLA (Independent Testing Laboratory Association), New England.

Watson, Kim. April, 2004. The New NELAC and ELAB Update. Presented to SQA Annual Meeting.

Watson, Kim & SQA EPA GLP Specialty Group. February 2005. GLP Inspection Experiences from the Regulated Communities Perspective. 2005 SQA World Conference.

ATTACHMENT F: MATERIAL SAFETY DATA SHEETS (MSDS)

1. 1,1,2,2-tetrachloroethane
2. 1,1,2-trichloroethane
3. 1,2-dichloroethane
4. 1,1-dichloroethene
5. Cis and Trans-1,2-dichloroethene
6. Trichloroethylene
7. Tetrachloroethylene
8. Vinyl Chloride
9. Alconox
10. Regular Unleaded Gasoline
11. Diesel Fuel
12. Methanol
13. Hydrochloric Acid
14. Conductivity Standard Solutions
15. Buffer Standard pH 7.00
16. Buffer Standard pH 10.00
17. Buffer Standard pH 4.00
18. AMCO Clear® 1.0 NTU Turbidity Calibration Standard
19. AMCO Clear® 10 NTU Turbidity Calibration Standard
20. AMCO Clear® 100 NTU Turbidity Calibration Standard
21. 100 PPM isobutylene in Air



Health	2
Fire	1
Reactivity	0
Personal Protection	H

Material Safety Data Sheet
1,1,2,2-Tetrachloroethane MSDS

Section 1: Chemical Product and Company Identification

Product Name: 1,1,2,2-Tetrachloroethane

Catalog Codes: SLT1927

CAS#: 79-34-5

RTECS: KI8575000

TSCA: TSCA 8(b) inventory: 1,1,2,2-Tetrachloroethane

CI#: Not available.

Synonym:

Chemical Name: Not available.

Chemical Formula: C2H2Cl4

Contact Information:

Sciencelab.com, Inc.
14025 Smith Rd.
Houston, Texas 77396

US Sales: **1-800-901-7247**
International Sales: **1-281-441-4400**

Order Online: ScienceLab.com

CHEMTREC (24HR Emergency Telephone), call:
1-800-424-9300

International CHEMTREC, call: 1-703-527-3887

For non-emergency assistance, call: 1-281-441-4400

Section 2: Composition and Information on Ingredients

Composition:

Name	CAS #	% by Weight
{1,1,2,2-}Tetrachloroethane	79-34-5	100

Toxicological Data on Ingredients: 1,1,2,2-Tetrachloroethane: ORAL (LD50): Acute: 250 mg/kg [Rat]. DERMAL (LD50): Acute: 6400 mg/kg [Rabbit]. VAPOR (LC50): Acute: 2250 ppm 4 hour(s) [Mouse].

Section 3: Hazards Identification

Potential Acute Health Effects:
Very hazardous in case of eye contact (irritant), of ingestion, of inhalation. Hazardous in case of skin contact (irritant, permeator). Inflammation of the eye is characterized by redness, watering, and itching.

Potential Chronic Health Effects:
CARCINOGENIC EFFECTS: Classified + (PROVEN) by OSHA. Classified 2 (Reasonably anticipated.) by NTP. MUTAGENIC EFFECTS: Not available. TERATOGENIC EFFECTS: Not available. DEVELOPMENTAL TOXICITY: Not available. The substance is toxic to blood, kidneys, the nervous system, liver. Repeated or prolonged exposure to the substance can produce target organs damage.

Section 4: First Aid Measures

Eye Contact:
Check for and remove any contact lenses. Immediately flush eyes with running water for at least 15 minutes, keeping eyelids open. Cold water may be used. Do not use an eye ointment. Seek medical attention.

Skin Contact:
After contact with skin, wash immediately with plenty of water. Gently and thoroughly wash the contaminated skin with running water and non-abrasive soap. Be particularly careful to clean folds, crevices, creases and groin. Cover the irritated skin with an emollient. If irritation persists, seek medical attention. Wash contaminated clothing before reusing.

Serious Skin Contact:
Wash with a disinfectant soap and cover the contaminated skin with an anti-bacterial cream. Seek immediate medical attention.

Inhalation: Allow the victim to rest in a well ventilated area. Seek immediate medical attention.

Serious Inhalation:
Evacuate the victim to a safe area as soon as possible. Loosen tight clothing such as a collar, tie, belt or waistband. If breathing is difficult, administer oxygen. If the victim is not breathing, perform mouth-to-mouth resuscitation. WARNING: It may be hazardous to the person providing aid to give mouth-to-mouth resuscitation when the inhaled material is toxic, infectious or corrosive. Seek medical attention.

Ingestion:
Do not induce vomiting. Examine the lips and mouth to ascertain whether the tissues are damaged, a possible indication that the toxic material was ingested; the absence of such signs, however, is not conclusive. Loosen tight clothing such as a collar, tie, belt or waistband. If the victim is not breathing, perform mouth-to-mouth resuscitation. Seek immediate medical attention.

Serious Ingestion: Not available.

Section 5: Fire and Explosion Data

Flammability of the Product: May be combustible at high temperature.

Auto-Ignition Temperature: Not available.

Flash Points: Not available.

Flammable Limits: Not available.

Products of Combustion: These products are carbon oxides (CO, CO2), halogenated compounds.

Fire Hazards in Presence of Various Substances: Not available.

Explosion Hazards in Presence of Various Substances:
Risks of explosion of the product in presence of mechanical impact: Not available. Risks of explosion of the product in presence of static discharge: Not available.

Fire Fighting Media and Instructions:
SMALL FIRE: Use DRY chemical powder. LARGE FIRE: Use water spray, fog or foam. Do not use water jet.

Special Remarks on Fire Hazards: Not available.

Special Remarks on Explosion Hazards: Not available.

Section 6: Accidental Release Measures

Small Spill: Absorb with an inert material and put the spilled material in an appropriate waste disposal.

Large Spill:
Absorb with an inert material and put the spilled material in an appropriate waste disposal. Be careful that the product is not present at a concentration level above TLV. Check TLV on the MSDS and with local authorities.

Section 7: Handling and Storage

Precautions:
Keep locked up Keep away from heat. Keep away from sources of ignition. Empty containers pose a fire risk, evaporate the residue under a fume hood. Ground all equipment containing material. Do not ingest. Do not breathe gas/fumes/ vapour/spray. In case of insufficient ventilation, wear suitable respiratory equipment If ingested, seek medical advice immediately and show the container or the label. Avoid contact with skin and eyes

Storage:
Keep container dry. Keep in a cool place. Ground all equipment containing material. Carcinogenic, teratogenic or mutagenic materials should be stored in a separate locked safety storage cabinet or room.

Section 8: Exposure Controls/Personal Protection

Engineering Controls:
Provide exhaust ventilation or other engineering controls to keep the airborne concentrations of vapors below their respective threshold limit value. Ensure that eyewash stations and safety showers are proximal to the work-station location.

Personal Protection:
Splash goggles. Lab coat. Vapor respirator. Be sure to use an approved/certified respirator or equivalent. Gloves.

Personal Protection in Case of a Large Spill:
Splash goggles. Full suit. Vapor respirator. Boots. Gloves. A self contained breathing apparatus should be used to avoid inhalation of the product. Suggested protective clothing might not be sufficient; consult a specialist BEFORE handling this product.

Exposure Limits:
TWA: 1 (ppm) from ACGIH (TLV) SKIN TWA: 6.9 (mg/m3) from ACGIH SKIN Consult local authorities for acceptable exposure limits.

Section 9: Physical and Chemical Properties

Physical state and appearance: Liquid.

Odor: Not available.

Taste: Not available.

Molecular Weight: 167.86 g/mole

Color: Not available.

pH (1% soln/water): Not available.

Boiling Point: 146.5°C (295.7°F)

Melting Point: -44°C (-47.2°F)

Critical Temperature: Not available.

Specific Gravity: 1.5866 (Water = 1)

Vapor Pressure: 8 mm of Hg (@ 20°C)

Vapor Density: 5.77 (Air = 1)

Volatility: Not available.

Odor Threshold: 3 ppm

Water/Oil Dist. Coeff.: The product is equally soluble in oil and water; log(oil/water) = 0

Ionicity (in Water): Not available.

Dispersion Properties: Not available.

Solubility: Very slightly soluble in cold water.

Section 10: Stability and Reactivity Data

Stability: The product is stable.

Instability Temperature: Not available.

Conditions of Instability: Not available.

Incompatibility with various substances: Not available.

Corrosivity: Non-corrosive in presence of glass.

Special Remarks on Reactivity: Not available.

Special Remarks on Corrosivity: Not available.

Polymerization: No.

Section 11: Toxicological Information

Routes of Entry: Dermal contact. Eye contact. Inhalation. Ingestion.

Toxicity to Animals:
WARNING: THE LC50 VALUES HEREUNDER ARE ESTIMATED ON THE BASIS OF A 4-HOUR EXPOSURE. Acute oral toxicity (LD50): 250 mg/kg [Rat]. Acute dermal toxicity (LD50): 6400 mg/kg [Rabbit]. Acute toxicity of the vapor (LC50): 2250 ppm 4 hour(s) [Mouse].

Chronic Effects on Humans:
CARCINOGENIC EFFECTS: Classified + (PROVEN) by OSHA. Classified 2 (Reasonably anticipated.) by NTP. The substance is toxic to blood, kidneys, the nervous system, liver.

Other Toxic Effects on Humans:
Very hazardous in case of ingestion, of inhalation. Hazardous in case of skin contact (irritant, permeator).

Special Remarks on Toxicity to Animals: Not available.

Special Remarks on Chronic Effects on Humans: Not available.

Special Remarks on other Toxic Effects on Humans: Not available.

Section 12: Ecological Information

Ecotoxicity: Not available.

BOD5 and COD: Not available.

Products of Biodegradation:
Possibly hazardous short term degradation products are not likely. However, long term degradation products may arise.

Toxicity of the Products of Biodegradation: The products of degradation are more toxic.

Special Remarks on the Products of Biodegradation: Not available.

Section 13: Disposal Considerations

Waste Disposal:

Section 14: Transport Information

DOT Classification: CLASS 6.1: Poisonous material.

Identification: : Tetrachloroethane : UN1702 PG: II

Special Provisions for Transport: Marine Pollutant

Section 15: Other Regulatory Information

Federal and State Regulations:
California prop. 65: This product contains the following ingredients for which the State of California has found to cause cancer, birth defects or other reproductive harm, which would require a warning under the statute: 1,1,2,2-Tetrachloroethane California prop. 65: This product contains the following ingredients for which the State of California has found to cause cancer which would require a warning under the statute: 1,1,2,2-Tetrachloroethane Pennsylvania RTK: 1,1,2,2-Tetrachloroethane Massachusetts RTK: 1,1,2,2-Tetrachloroethane TSCA 8(b) inventory: 1,1,2,2-Tetrachloroethane SARA 313 toxic chemical notification and release reporting: 1,1,2,2-Tetrachloroethane CERCLA: Hazardous substances.: 1,1,2,2-Tetrachloroethane

Other Regulations: OSHA: Hazardous by definition of Hazard Communication Standard (29 CFR 1910.1200).

Other Classifications:

WHMIS (Canada): CLASS D-1A: Material causing immediate and serious toxic effects (VERY TOXIC).

DSCL (EEC):
R38- Irritating to skin. R41- Risk of serious damage to eyes. R45- May cause cancer.

HMIS (U.S.A.):

Health Hazard: 2

Fire Hazard: 1

Reactivity: 0

Personal Protection: h

National Fire Protection Association (U.S.A.):

Health: 2

Flammability: 1

Reactivity: 0

Specific hazard:

Protective Equipment:
Gloves. Lab coat. Vapor respirator. Be sure to use an approved/certified respirator or equivalent. Wear appropriate respirator when ventilation is inadequate. Splash goggles.

Section 16: Other Information

References: Not available.

Other Special Considerations: Not available.

Created: 10/10/2005 12:01 AM

Last Updated: 11/01/2010 12:00 PM

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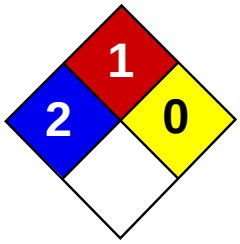

Health	2
Fire	1
Reactivity	0
Personal Protection	J

Material Safety Data Sheet
1,1,2-Trichloroethane MSDS

Section 1: Chemical Product and Company Identification

Product Name: 1,1,2-Trichloroethane

Catalog Codes: SLT1450

CAS#: 79-00-5

RTECS: KJ3150000

TSCA: TSCA 8(b) inventory: 1,1,2-Trichloroethane

CI#: Not applicable.

Synonym: beta-T; beta-Trichloroethane

Chemical Name: 1,1,2-Trichloroethane

Chemical Formula: C2H3Cl3

Contact Information:

Sciencelab.com, Inc.
14025 Smith Rd.
Houston, Texas 77396

US Sales: **1-800-901-7247**
International Sales: **1-281-441-4400**

Order Online: ScienceLab.com

CHEMTREC (24HR Emergency Telephone), call:
1-800-424-9300

International CHEMTREC, call: 1-703-527-3887

For non-emergency assistance, call: 1-281-441-4400

Section 2: Composition and Information on Ingredients

Composition:

Name	CAS #	% by Weight
{1,1,2-}Trichloroethane	79-00-5	100

Toxicological Data on Ingredients: 1,1,2-Trichloroethane: ORAL (LD50): Acute: 836 mg/kg [Rat]. 378 mg/kg [Mouse]. DERMAL (LD50): Acute: 5377 mg/kg [Rabbit].

Section 3: Hazards Identification

Potential Acute Health Effects:
Very hazardous in case of eye contact (irritant). Hazardous in case of skin contact (irritant). Inflammation of the eye is characterized by redness, watering, and itching.

Potential Chronic Health Effects:
CARCINOGENIC EFFECTS: 3 (Not classifiable for human.) by IARC. MUTAGENIC EFFECTS: Mutagenic for mammalians. Mutagenic for bacteria and/or yeast. TERATOGENIC EFFECTS: Not available. DEVELOPMENTAL TOXICITY: Not available. The substance is toxic to lungs, the nervous system, liver, brain, digestive system, gastrointestinal tract, endocrine. Repeated or prolonged exposure to the substance can produce target organs damage.

Section 4: First Aid Measures

Eye Contact:

Check for and remove any contact lenses. Immediately flush eyes with running water for at least 15 minutes, keeping eyelids open. Cold water may be used. Do not use an eye ointment. Seek medical attention.

Skin Contact:
After contact with skin, wash immediately with plenty of water. Gently and thoroughly wash the contaminated skin with running water and non-abrasive soap. Be particularly careful to clean folds, crevices, creases and groin. Cover the irritated skin with an emollient. If irritation persists, seek medical attention. Wash contaminated clothing before reusing.

Serious Skin Contact:
Wash with a disinfectant soap and cover the contaminated skin with an anti-bacterial cream. Seek immediate medical attention.

Inhalation: Allow the victim to rest in a well ventilated area. Seek immediate medical attention.

Serious Inhalation:
Evacuate the victim to a safe area as soon as possible. Loosen tight clothing such as a collar, tie, belt or waistband. If breathing is difficult, administer oxygen. If the victim is not breathing, perform mouth-to-mouth resuscitation. Seek medical attention.

Ingestion:
Do not induce vomiting. Examine the lips and mouth to ascertain whether the tissues are damaged, a possible indication that the toxic material was ingested; the absence of such signs, however, is not conclusive. Loosen tight clothing such as a collar, tie, belt or waistband. If the victim is not breathing, perform mouth-to-mouth resuscitation. Seek immediate medical attention.

Serious Ingestion: Not available.

Section 5: Fire and Explosion Data

Flammability of the Product: May be combustible at high temperature.

Auto-Ignition Temperature: 459°C (858.2°F)

Flash Points: Not available.

Flammable Limits: LOWER: 6% UPPER: 15.5%

Products of Combustion: These products are carbon oxides (CO, CO2), halogenated compounds.

Fire Hazards in Presence of Various Substances: Slightly flammable to flammable in presence of open flames and sparks.

Explosion Hazards in Presence of Various Substances:
Risks of explosion of the product in presence of mechanical impact: Not available. Risks of explosion of the product in presence of static discharge: Not available.

Fire Fighting Media and Instructions:
SMALL FIRE: Use DRY chemical powder. LARGE FIRE: Use water spray, fog or foam. Do not use water jet.

Special Remarks on Fire Hazards: Not available.

Special Remarks on Explosion Hazards: Not available.

Section 6: Accidental Release Measures

Small Spill: Absorb with an inert material and put the spilled material in an appropriate waste disposal.

Large Spill:
Absorb with an inert material and put the spilled material in an appropriate waste disposal. Be careful that the product is not present at a concentration level above TLV. Check TLV on the MSDS and with local authorities.

Section 7: Handling and Storage

Precautions:

Keep locked up Keep away from heat. Keep away from sources of ignition. Empty containers pose a fire risk, evaporate the residue under a fume hood. Ground all equipment containing material. Do not ingest. Do not breathe gas/fumes/ vapour/spray. If ingested, seek medical advice immediately and show the container or the label. Avoid contact with skin and eyes

Storage:
Keep container dry. Keep in a cool place. Ground all equipment containing material. Keep container tightly closed. Keep in a cool, well-ventilated place. Combustible materials should be stored away from extreme heat and away from strong oxidizing agents.

Section 8: Exposure Controls/Personal Protection

Engineering Controls:
Provide exhaust ventilation or other engineering controls to keep the airborne concentrations of vapors below their respective threshold limit value. Ensure that eyewash stations and safety showers are proximal to the work-station location.

Personal Protection: Splash goggles. Lab coat. Gloves.

Personal Protection in Case of a Large Spill:
Splash goggles. Full suit. Boots. Gloves. Suggested protective clothing might not be sufficient; consult a specialist BEFORE handling this product.

Exposure Limits:
USA: TWA: 10 (ppm) from OSHA (PEL) SKIN TWA: 55 (mg/m3) from ACGIH SKIN Consult local authorities for acceptable exposure limits.

Section 9: Physical and Chemical Properties

Physical state and appearance: Liquid. (Clear)

Odor: Sweet chlorofor like

Taste: Not available.

Molecular Weight: 133.41 g/mole

Color: Colorless.

pH (1% soln/water): Not available.

Boiling Point: 113.8°C (236.8°F)

Melting Point: -36.6°C (-33.9°F)

Critical Temperature: Not available.

Specific Gravity: 1.4416 (Water = 1)

Vapor Pressure: Not available.

Vapor Density: 4.63 (Air = 1)

Volatility: Not available.

Odor Threshold: Not available.

Water/Oil Dist. Coeff.: The product is more soluble in oil; log(oil/water) = 2.2

Ionicity (in Water): Not available.

Dispersion Properties:
Partially dispersed in methanol, diethyl ether. See solubility in water, methanol, diethyl ether, acetone.

Solubility:
Partially soluble in methanol, diethyl ether, acetone. Very slightly soluble in cold water.

Section 10: Stability and Reactivity Data

Stability: The product is stable.

Instability Temperature: Not available.

Conditions of Instability: Not available.

Incompatibility with various substances: Slightly reactive to reactive with oxidizing agents, metals, alkalis.

Corrosivity: Corrosive in presence of aluminum, of zinc.

Special Remarks on Reactivity: Not available.

Special Remarks on Corrosivity: Not available.

Polymerization: No.

Section 11: Toxicological Information

Routes of Entry: Absorbed through skin. Dermal contact. Eye contact. Inhalation. Ingestion.

Toxicity to Animals:
Acute oral toxicity (LD50): 378 mg/kg [Mouse]. Acute dermal toxicity (LD50): 5377 mg/kg [Rabbit].

Chronic Effects on Humans:
CARCINOGENIC EFFECTS: 3 (Not classifiable for human.) by IARC. MUTAGENIC EFFECTS: Mutagenic for mammalians. Mutagenic for bacteria and/or yeast. The substance is toxic to lungs, the nervous system, liver, brain, digestive system, gastrointestinal tract, endocrine.

Other Toxic Effects on Humans: Hazardous in case of skin contact (irritant).

Special Remarks on Toxicity to Animals: Not available.

Special Remarks on Chronic Effects on Humans: Not available.

Special Remarks on other Toxic Effects on Humans: Not available.

Section 12: Ecological Information

Ecotoxicity: Not available.

BOD5 and COD: Not available.

Products of Biodegradation:
Possibly hazardous short term degradation products are not likely. However, long term degradation products may arise.

Toxicity of the Products of Biodegradation: The products of degradation are as toxic as the product itself.

Special Remarks on the Products of Biodegradation: Not available.

Section 13: Disposal Considerations

Waste Disposal:

Section 14: Transport Information

DOT Classification: CLASS 6.1: Poisonous material.

Identification: : Toxic liquids n.o.s. : UN2810 PG: Not available.

Special Provisions for Transport: Not available.

Section 15: Other Regulatory Information

Federal and State Regulations:
California prop. 65: This product contains the following ingredients for which the State of California has found to cause cancer, birth defects or other reproductive harm, which would require a warning under the statute: 1,1,2-Trichloroethane California prop. 65 (no significant risk level): 1,1,2-Trichloroethane: 0.01 mg/day (inhalation) California prop. 65: This product contains the following ingredients for which the State of California has found to cause cancer which would require a warning under the statute: 1,1,2-Trichloroethane Rhode Island RTK hazardous substances: 1,1,2-Trichloroethane Pennsylvania RTK: 1,1,2-Trichloroethane Florida: 1,1,2-Trichloroethane Minnesota: 1,1,2-Trichloroethane Michigan critical material: 1,1,2-Trichloroethane Massachusetts RTK: 1,1,2-Trichloroethane New Jersey: 1,1,2-Trichloroethane TSCA 8(b) inventory: 1,1,2-Trichloroethane TSCA 8(a) PAIR: 1,1,2-Trichloroethane SARA 313 toxic chemical notification and release reporting: 1,1,2-Trichloroethane CERCLA: Hazardous substances.: 1,1,2-Trichloroethane: 100 lbs. (45.36 kg)

Other Regulations: OSHA: Hazardous by definition of Hazard Communication Standard (29 CFR 1910.1200).

Other Classifications:

WHMIS (Canada): CLASS D-1B: Material causing immediate and serious toxic effects (TOXIC).

DSCL (EEC):
R22- Harmful if swallowed. R38- Irritating to skin. R41- Risk of serious damage to eyes.

HMIS (U.S.A.):

Health Hazard: 2

Fire Hazard: 1

Reactivity: 0

Personal Protection: j

National Fire Protection Association (U.S.A.):

Health: 2

Flammability: 1

Reactivity: 0

Specific hazard:

Protective Equipment:
Gloves. Lab coat. Wear appropriate respirator when ventilation is inadequate. Splash goggles.

Section 16: Other Information

References: Not available.

Other Special Considerations: Not available.

Created: 10/11/2005 12:48 PM

Last Updated: 11/01/2010 12:00 PM

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Material Safety Data Sheet

Version 4.3
Revision Date 01/19/2012
Print Date 07/10/2012

1. PRODUCT AND COMPANY IDENTIFICATION

Product name	:	1,1-Dichloroethene
Product Number	:	48526
Brand	:	Supelco
Supplier	:	Sigma-Aldrich 3050 Spruce Street SAINT LOUIS MO 63103 USA
Telephone	:	+1 800-325-5832
Fax	:	+1 800-325-5052
Emergency Phone # (For both supplier and manufacturer)	:	(314) 776-6555
Preparation Information	:	Sigma-Aldrich Corporation Product Safety - Americas Region 1-800-521-8956

2. HAZARDS IDENTIFICATION

Emergency Overview

OSHA Hazards
Flammable liquid, Target Organ Effect, Toxic by ingestion, Irritant, Carcinogen

Target Organs

Liver, Kidney, Central nervous system

GHS Classification
Flammable liquids (Category 1)
Acute toxicity, Oral (Category 3)
Skin irritation (Category 2)
Eye irritation (Category 2A)
Carcinogenicity (Category 2)

GHS Label elements, including precautionary statements

Pictogram

Signal word Danger

Hazard statement(s)

H224	Extremely flammable liquid and vapour.
H301	Toxic if swallowed.
H315	Causes skin irritation.
H319	Causes serious eye irritation.
H351	Suspected of causing cancer.

Precautionary statement(s)

P210	Keep away from heat/sparks/open flames/hot surfaces. - No smoking.
P281	Use personal protective equipment as required.
P301 + P310	IF SWALLOWED: Immediately call a POISON CENTER or doctor/ physician.
P305 + P351 + P338	IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing.

HMIS Classification

Health hazard:	2
Chronic Health Hazard:	*
Flammability:	4
Physical hazards:	2

NFPA Rating

Health hazard:	2
Fire:	4
Reactivity Hazard:	0

Potential Health Effects

Inhalation	May be harmful if inhaled. Causes respiratory tract irritation.
Skin	May be harmful if absorbed through skin. Causes skin irritation.
Eyes	Causes eye irritation.
Ingestion	Toxic if swallowed.

3. COMPOSITION/INFORMATION ON INGREDIENTS

Synonyms : 1,1-Dichloroethylene
Vinylidene chloride

Formula : $C_2H_2Cl_2$

Molecular Weight : 96.94 g/mol

Component		Concentration
Vinylidene chloride		
CAS-No.	75-35-4	-
EC-No.	200-864-0	
Index-No.	602-025-00-8	

4. FIRST AID MEASURES

General advice
Consult a physician. Show this safety data sheet to the doctor in attendance.Move out of dangerous area.

If inhaled
If breathed in, move person into fresh air. If not breathing, give artificial respiration. Consult a physician.

In case of skin contact
Wash off with soap and plenty of water. Take victim immediately to hospital. Consult a physician.

In case of eye contact
Rinse thoroughly with plenty of water for at least 15 minutes and consult a physician.

If swallowed
Do NOT induce vomiting. Never give anything by mouth to an unconscious person. Rinse mouth with water. Consult a physician.

5. FIREFIGHTING MEASURES

Conditions of flammability
Flammable in the presence of a source of ignition when the temperature is above the flash point. Keep away from heat/sparks/open flame/hot surface. No smoking.

Suitable extinguishing media
Use water spray, alcohol-resistant foam, dry chemical or carbon dioxide.

Special protective equipment for firefighters
Wear self contained breathing apparatus for fire fighting if necessary.

Hazardous combustion products
Hazardous decomposition products formed under fire conditions. - Carbon oxides, Hydrogen chloride gas

Further information
Use water spray to cool unopened containers.

6. ACCIDENTAL RELEASE MEASURES

Personal precautions
Wear respiratory protection. Avoid breathing vapors, mist or gas. Ensure adequate ventilation. Remove all sources of ignition. Evacuate personnel to safe areas. Beware of vapours accumulating to form explosive concentrations. Vapours can accumulate in low areas.

Environmental precautions
Prevent further leakage or spillage if safe to do so. Do not let product enter drains.

Methods and materials for containment and cleaning up
Contain spillage, and then collect with an electrically protected vacuum cleaner or by wet-brushing and place in container for disposal according to local regulations (see section 13).

7. HANDLING AND STORAGE

Precautions for safe handling
Avoid contact with skin and eyes. Avoid inhalation of vapour or mist.
Use explosion-proof equipment. Keep away from sources of ignition - No smoking. Take measures to prevent the build up of electrostatic charge.

Conditions for safe storage
Keep container tightly closed in a dry and well-ventilated place. Containers which are opened must be carefully resealed and kept upright to prevent leakage.

Air and moisture sensitive. Store under inert gas.

8. EXPOSURE CONTROLS/PERSONAL PROTECTION

Components with workplace control parameters

Components	CAS-No.	Value	Control parameters	Basis
Remarks	Potential Occupational Carcinogen See Appendix A			
Vinylidene chloride	75-35-4	TWA	5 ppm	USA. ACGIH Threshold Limit Values (TLV)
	Liver & kidney damage Not classifiable as a human carcinogen			
		TWA	1 ppm 4 mg/m3	USA. OSHA - TABLE Z-1 Limits for Air Contaminants - 1910.1000

Personal protective equipment

Respiratory protection
Where risk assessment shows air-purifying respirators are appropriate use a full-face respirator with multi-purpose combination (US) or type AXBEK (EN 14387) respirator cartridges as a backup to engineering controls. If the respirator is the sole means of protection, use a full-face supplied air respirator. Use respirators and components tested and approved under appropriate government standards such as NIOSH (US) or CEN (EU).

Hand protection
Handle with gloves. Gloves must be inspected prior to use. Use proper glove removal technique (without touching glove's outer surface) to avoid skin contact with this product. Dispose of contaminated gloves after use in accordance with applicable laws and good laboratory practices. Wash and dry hands.

Eye protection
Face shield and safety glasses Use equipment for eye protection tested and approved under appropriate government standards such as NIOSH (US) or EN 166(EU).

Skin and body protection

Complete suit protecting against chemicals, Flame retardant antistatic protective clothing, The type of protective equipment must be selected according to the concentration and amount of the dangerous substance at the specific workplace.

Hygiene measures
Avoid contact with skin, eyes and clothing. Wash hands before breaks and immediately after handling the product.

9. PHYSICAL AND CHEMICAL PROPERTIES

Appearance

Form	liquid, clear
Colour	colourless

Safety data

pH	no data available
Melting point/freezing point	Melting point/range: -122 °C (-188 °F) - lit.
Boiling point	30 - 32 °C (86 - 90 °F) - lit.
Flash point	-25.0 °C (-13.0 °F) - closed cup
Ignition temperature	520 °C (968 °F)
Autoignition temperature	520.0 °C (968.0 °F) 580.0 °C (1,076.0 °F)
Lower explosion limit	6.5 %(V)
Upper explosion limit	15.5 %(V)
Vapour pressure	658.6 hPa (494.0 mmHg) 667.3 hPa (500.5 mmHg) at 20.0 °C (68.0 °F) 2,137.4 hPa (1,603.2 mmHg) at 55.0 °C (131.0 °F)
Density	1.213 g/cm3 at 20 °C (68 °F)
Water solubility	0.2 g/l at 20 °C (68 °F)
Partition coefficient: n-octanol/water	no data available
Relative vapour density	no data available
Odour	no data available
Odour Threshold	no data available
Evaporation rate	no data available

10. STABILITY AND REACTIVITY

Chemical stability
Stable under recommended storage conditions.

Possibility of hazardous reactions

Vapours may form explosive mixture with air.

Conditions to avoid
Heat, flames and sparks. Extremes of temperature and direct sunlight.

Materials to avoid
Oxidizing agents, Copper, Aluminum, and its alloys, Peroxides, Strong bases, Oxygen

Hazardous decomposition products
Hazardous decomposition products formed under fire conditions. - Carbon oxides, Hydrogen chloride gas
Other decomposition products - no data available

11. TOXICOLOGICAL INFORMATION

Acute toxicity

Oral LD50
LD50 Oral - rat - 200.0 mg/kg

Inhalation LC50
Lung irritation

Dermal LD50
no data available

Other information on acute toxicity
no data available

Skin corrosion/irritation
no data available

Serious eye damage/eye irritation
no data available

Respiratory or skin sensitization
no data available

Germ cell mutagenicity
Laboratory experiments have shown mutagenic effects.

Carcinogenicity

This product is or contains a component that has been reported to be possibly carcinogenic based on its IARC, ACGIH, NTP, or EPA classification.

Limited evidence of carcinogenicity in animal studies

IARC:	3 - Group 3: Not classifiable as to its carcinogenicity to humans (Vinylidene chloride)
NTP:	No component of this product present at levels greater than or equal to 0.1% is identified as a known or anticipated carcinogen by NTP.
OSHA:	No component of this product present at levels greater than or equal to 0.1% is identified as a carcinogen or potential carcinogen by OSHA.

Reproductive toxicity

no data available

Teratogenicity

no data available

Specific target organ toxicity - single exposure (Globally Harmonized System)

no data available

Specific target organ toxicity - repeated exposure (Globally Harmonized System)
no data available

Aspiration hazard
no data available

Potential health effects

Inhalation	May be harmful if inhaled. Causes respiratory tract irritation.
Ingestion	Toxic if swallowed.
Skin	May be harmful if absorbed through skin. Causes skin irritation.
Eyes	Causes eye irritation.

Signs and Symptoms of Exposure
Nausea, Headache, Vomiting, Dizziness, Drowsiness, Confusion., Incoordination., Central nervous system depression, To the best of our knowledge, the chemical, physical, and toxicological properties have not been thoroughly investigated.

Synergistic effects
no data available

Additional Information
RTECS: KV9275000

12. ECOLOGICAL INFORMATION

Toxicity

Toxicity to fish	LC50 - Daphnia magna (Water flea) - 11.60 - 11.79 mg/l
	LC50 - Pimephales promelas (fathead minnow) - 108.00 - 169.00 mg/l
	LC50 - Lepomis macrochirus (Bluegill) - 74.00 - 220.00 mg/l
	LC50 - Cyprinodon variegatus (sheepshead minnow) - 249.00 mg/l
	LC50 - other fish - 250.00 mg/l
	LC50 - other fish - 224.00 mg/l
	LC50 - Pimephales promelas (fathead minnow) - 108 mg/l - 96 h
	NOEC - Cyprinodon variegatus (sheepshead minnow) - 80 mg/l - 96 h
Toxicity to daphnia and other aquatic invertebrates	LC50 - Daphnia magna (Water flea) - 11.6 mg/l - 48 h

Persistence and degradability
no data available

Bioaccumulative potential
no data available

Mobility in soil
no data available

PBT and vPvB assessment
no data available

Other adverse effects

no data available

13. DISPOSAL CONSIDERATIONS

Product
Burn in a chemical incinerator equipped with an afterburner and scrubber but exert extra care in igniting as this material is highly flammable. Offer surplus and non-recyclable solutions to a licensed disposal company. Contact a licensed professional waste disposal service to dispose of this material.

Contaminated packaging
Dispose of as unused product.

14. TRANSPORT INFORMATION

DOT (US)
UN number: 1303 Class: 3 Packing group: I
Proper shipping name: Vinylidene chloride, stabilized
Reportable Quantity (RQ): 100 lbs
Marine pollutant: No
Poison Inhalation Hazard: No

IMDG
UN number: 1303 Class: 3 Packing group: I EMS-No: F-E, S-D
Proper shipping name: VINYLIDENE CHLORIDE, STABILIZED
Marine pollutant: Marine pollutant

IATA
UN number: 1303 Class: 3 Packing group: I
Proper shipping name: Vinylidene chloride, stabilized

15. REGULATORY INFORMATION

OSHA Hazards
Flammable liquid, Target Organ Effect, Toxic by ingestion, Irritant, Carcinogen

SARA 302 Components
SARA 302: No chemicals in this material are subject to the reporting requirements of SARA Title III, Section 302.

SARA 313 Components
The following components are subject to reporting levels established by SARA Title III, Section 313:

	CAS-No.	Revision Date
Vinylidene chloride	75-35-4	2007-07-01

SARA 311/312 Hazards
Fire Hazard, Acute Health Hazard, Chronic Health Hazard

Massachusetts Right To Know Components

	CAS-No.	Revision Date
Vinylidene chloride	75-35-4	2007-07-01

Pennsylvania Right To Know Components

	CAS-No.	Revision Date
Vinylidene chloride	75-35-4	2007-07-01

New Jersey Right To Know Components

	CAS-No.	Revision Date
Vinylidene chloride	75-35-4	2007-07-01

California Prop. 65 Components
This product does not contain any chemicals known to State of California to cause cancer, birth defects, or any other reproductive harm.

16. OTHER INFORMATION

Further information
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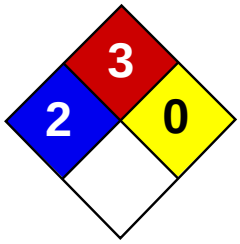
The above information is believed to be correct but does not purport to be all inclusive and shall be used only as a guide. The information in this document is based on the present state of our knowledge and is applicable to the product with regard to appropriate safety precautions. It does not represent any guarantee of the properties of the product. Sigma-Aldrich Corporation and its Affiliates shall not be held liable for any damage resulting from handling or from contact with the above product. See www.sigma-aldrich.com and/or the reverse side of invoice or packing slip for additional terms and conditions of sale.



Health	2
Fire	3
Reactivity	0
Personal Protection	H

Material Safety Data Sheet
1,2-Dichloroethane MSDS

Section 1: Chemical Product and Company Identification

Product Name: 1,2-Dichloroethane

Catalog Codes: SLD2521, SLD3721

CAS#: 107-06-2

RTECS: KH9800000

TSCA: TSCA 8(b) inventory: 1,2-Dichloroethane

CI#: Not available.

Synonym: Ethylene dichloride

Chemical Formula: C2H4CL2

Contact Information:

Sciencelab.com, Inc.
14025 Smith Rd.
Houston, Texas 77396

US Sales: **1-800-901-7247**
International Sales: **1-281-441-4400**

Order Online: ScienceLab.com

CHEMTREC (24HR Emergency Telephone), call: 1-800-424-9300

International CHEMTREC, call: 1-703-527-3887

For non-emergency assistance, call: 1-281-441-4400

Section 2: Composition and Information on Ingredients

Composition:

Name	CAS #	% by Weight
{1,2-}Dichloroethane	107-06-2	100

Toxicological Data on Ingredients: 1,2-Dichloroethane: ORAL (LD50): Acute: 670 mg/kg [Rat]. 413 mg/kg [Mouse]. DERMAL (LD50): Acute: 2800 mg/kg [Rabbit]. VAPOR (LC50): Acute: 1414.2 ppm 4 hour(s) [Rat].

Section 3: Hazards Identification

Potential Acute Health Effects:
Extremely hazardous in case of ingestion. Very hazardous in case of eye contact (irritant), of inhalation. Hazardous in case of skin contact (irritant). Corrosive to skin and eyes on contact. Liquid or spray mist may produce tissue damage particularly on mucous membranes of eyes, mouth and respiratory tract. Skin contact may produce burns. Inhalation of the spray mist may produce severe irritation of respiratory tract, characterized by coughing, choking, or shortness of breath. Inflammation of the eye is characterized by redness, watering, and itching.

Potential Chronic Health Effects:
Very hazardous in case of ingestion, of inhalation. CARCINOGENIC EFFECTS: Classified + (PROVEN) by OSHA. Classified 2B (Possible for human.) by IARC. Classified 2 (Reasonably anticipated.) by NTP. MUTAGENIC EFFECTS: Not available. TERATOGENIC EFFECTS: Not available. DEVELOPMENTAL TOXICITY: Not available. The substance is toxic to lungs, the nervous system, liver, mucous membranes. Repeated or prolonged exposure to the substance can produce target organs damage. Repeated or prolonged contact with spray mist may produce chronic eye irritation and severe skin irritation. Repeated or prolonged exposure to spray mist may produce respiratory tract irritation leading to frequent attacks of bronchial infection.

Section 4: First Aid Measures

Eye Contact:
Check for and remove any contact lenses. Immediately flush eyes with running water for at least 15 minutes, keeping eyelids open. Cold water may be used. Do not use an eye ointment. Seek medical attention.

Skin Contact:
If the chemical got onto the clothed portion of the body, remove the contaminated clothes as quickly as possible, protecting your own hands and body. Place the victim under a deluge shower. If the chemical got on the victim's exposed skin, such as the hands : Gently and thoroughly wash the contaminated skin with running water and non-abrasive soap. Be particularly careful to clean folds, crevices, creases and groin. If irritation persists, seek medical attention. Wash contaminated clothing before reusing.

Serious Skin Contact:
Wash with a disinfectant soap and cover the contaminated skin with an anti-bacterial cream. Seek immediate medical attention.

Inhalation: Allow the victim to rest in a well ventilated area. Seek immediate medical attention.

Serious Inhalation:
Evacuate the victim to a safe area as soon as possible. Loosen tight clothing such as a collar, tie, belt or waistband. If breathing is difficult, administer oxygen. If the victim is not breathing, perform mouth-to-mouth resuscitation. WARNING: It may be hazardous to the person providing aid to give mouth-to-mouth resuscitation when the inhaled material is toxic, infectious or corrosive. Seek immediate medical attention.

Ingestion:
Do not induce vomiting. Examine the lips and mouth to ascertain whether the tissues are damaged, a possible indication that the toxic material was ingested; the absence of such signs, however, is not conclusive. Loosen tight clothing such as a collar, tie, belt or waistband. If the victim is not breathing, perform mouth-to-mouth resuscitation. Seek immediate medical attention.

Serious Ingestion: Not available.

Section 5: Fire and Explosion Data

Flammability of the Product: Flammable.

Auto-Ignition Temperature: 413°C (775.4°F)

Flash Points: CLOSED CUP: 13°C (55.4°F). OPEN CUP: 18°C (64.4°F).

Flammable Limits: LOWER: 6.2% UPPER: 15.6%

Products of Combustion: These products are carbon oxides (CO, CO2).

Fire Hazards in Presence of Various Substances:
Flammable in presence of open flames and sparks. Slightly flammable to flammable in presence of oxidizing materials.

Explosion Hazards in Presence of Various Substances:
Risks of explosion of the product in presence of mechanical impact: Not available. Risks of explosion of the product in presence of static discharge: Not available. Slightly explosive to explosive in presence of oxidizing materials.

Fire Fighting Media and Instructions:
Flammable liquid, soluble or dispersed in water. SMALL FIRE: Use DRY chemical powder. LARGE FIRE: Use alcohol foam, water spray or fog.

Special Remarks on Fire Hazards: Not available.

Special Remarks on Explosion Hazards: Not available.

Section 6: Accidental Release Measures

Small Spill: Absorb with an inert material and put the spilled material in an appropriate waste disposal.

Large Spill:
Flammable liquid. Corrosive liquid. Keep away from heat. Keep away from sources of ignition. Stop leak if without risk. Absorb with DRY earth, sand or other non-combustible material. Do not get water inside container. Do not touch spilled material. Use water spray curtain to divert vapor drift. Prevent entry into sewers, basements or confined areas; dike if needed. Eliminate all ignition sources. Call for assistance on disposal. Be careful that the product is not present at a concentration level above TLV. Check TLV on the MSDS and with local authorities.

Section 7: Handling and Storage

Precautions:
Keep locked up Keep container dry. Keep away from heat. Keep away from sources of ignition. Ground all equipment containing material. Do not ingest. Do not breathe gas/fumes/ vapour/spray. Never add water to this product In case of insufficient ventilation, wear suitable respiratory equipment If ingested, seek medical advice immediately and show the container or the label. Avoid contact with skin and eyes

Storage:
Flammable materials should be stored in a separate safety storage cabinet or room. Keep away from heat. Keep away from sources of ignition. Keep container tightly closed. Keep in a cool, well-ventilated place. Ground all equipment containing material. A refrigerated room would be preferable for materials with a flash point lower than 37.8°C (100°F).

Section 8: Exposure Controls/Personal Protection

Engineering Controls:
Provide exhaust ventilation or other engineering controls to keep the airborne concentrations of vapors below their respective threshold limit value. Ensure that eyewash stations and safety showers are proximal to the work-station location.

Personal Protection:
Splash goggles. Lab coat. Vapor respirator. Be sure to use an approved/certified respirator or equivalent. Gloves.

Personal Protection in Case of a Large Spill:
Splash goggles. Full suit. Vapor respirator. Boots. Gloves. A self contained breathing apparatus should be used to avoid inhalation of the product. Suggested protective clothing might not be sufficient; consult a specialist BEFORE handling this product.

Exposure Limits:
TWA: 10 CEIL: 75 (ppm) from ACGIH (TLV) TWA: 40 CEIL: 300 (mg/m3) from ACGIHConsult local authorities for acceptable exposure limits.

Section 9: Physical and Chemical Properties

Physical state and appearance: Liquid.

Odor: Not available.

Taste: Not available.

Molecular Weight: 98.96 g/mole

Color: Not available.

pH (1% soln/water): Not available.

Boiling Point: 83.5°C (182.3°F)

Melting Point: -35.3°C (-31.5°F)

Critical Temperature: Not available.

Specific Gravity: 1.2351 (Water = 1)

Vapor Pressure: 61 mm of Hg (@ 20°C)

Vapor Density: 3.42 (Air = 1)

Volatility: Not available.

Odor Threshold: 26 ppm

Water/Oil Dist. Coeff.: The product is equally soluble in oil and water; log(oil/water) = 0

Ionicity (in Water): Not available.

Dispersion Properties: See solubility in water, methanol, diethyl ether, n-octanol, acetone.

Solubility:
Easily soluble in methanol, diethyl ether, n-octanol, acetone. Very slightly soluble in cold water.

Section 10: Stability and Reactivity Data

Stability: The product is stable.

Instability Temperature: Not available.

Conditions of Instability: Not available.

Incompatibility with various substances: Not available.

Corrosivity: Non-corrosive in presence of glass.

Special Remarks on Reactivity: Not available.

Special Remarks on Corrosivity: Not available.

Polymerization: No.

Section 11: Toxicological Information

Routes of Entry: Eye contact. Inhalation. Ingestion.

Toxicity to Animals:
WARNING: THE LC50 VALUES HEREUNDER ARE ESTIMATED ON THE BASIS OF A 4-HOUR EXPOSURE. Acute oral toxicity (LD50): 413 mg/kg [Mouse]. Acute dermal toxicity (LD50): 2800 mg/kg [Rabbit]. Acute toxicity of the vapor (LC50): 1414.2 ppm 4 hour(s) [Rat].

Chronic Effects on Humans:
CARCINOGENIC EFFECTS: Classified + (PROVEN) by OSHA. Classified 2B (Possible for human.) by IARC. Classified 2 (Reasonably anticipated.) by NTP. The substance is toxic to lungs, the nervous system, liver, mucous membranes.

Other Toxic Effects on Humans:
Extremely hazardous in case of ingestion. Very hazardous in case of inhalation. Hazardous in case of skin contact (irritant).

Special Remarks on Toxicity to Animals: Not available.

Special Remarks on Chronic Effects on Humans: Passes through the placental barrier in animal. Excreted in maternal milk in human.

Special Remarks on other Toxic Effects on Humans: Not available.

Section 12: Ecological Information

Ecotoxicity: Not available.

BOD5 and COD: Not available.

Products of Biodegradation:
Possibly hazardous short term degradation products are not likely. However, long term degradation products may arise.

Toxicity of the Products of Biodegradation: The products of degradation are more toxic.

Special Remarks on the Products of Biodegradation: Not available.

Section 13: Disposal Considerations

Waste Disposal:

Section 14: Transport Information

DOT Classification: Class 3: Flammable liquid.

Identification: : Ethylene dichloride : UN1184 PG: II

Special Provisions for Transport: Marine Pollutant

Section 15: Other Regulatory Information

Federal and State Regulations:
California prop. 65: This product contains the following ingredients for which the State of California has found to cause cancer, birth defects or other reproductive harm, which would require a warning under the statute: 1,2-Dichloroethane California prop. 65: This product contains the following ingredients for which the State of California has found to cause cancer which would require a warning under the statute: 1,2-Dichloroethane Pennsylvania RTK: 1,2-Dichloroethane Massachusetts RTK: 1,2-Dichloroethane TSCA 8(b) inventory: 1,2-Dichloroethane CERCLA: Hazardous substances.: 1,2-Dichloroethane

Other Regulations: OSHA: Hazardous by definition of Hazard Communication Standard (29 CFR 1910.1200).

Other Classifications:

WHMIS (Canada):
CLASS B-2: Flammable liquid with a flash point lower than 37.8°C (100°F). CLASS D-1A: Material causing immediate and serious toxic effects (VERY TOXIC). CLASS D-2A: Material causing other toxic effects (VERY TOXIC). CLASS E: Corrosive liquid.

DSCL (EEC):
R11- Highly flammable. R20/22- Harmful by inhalation and if swallowed. R38- Irritating to skin. R41- Risk of serious damage to eyes. R45- May cause cancer.

HMIS (U.S.A.):

Health Hazard: 2

Fire Hazard: 3

Reactivity: 0

Personal Protection: h

National Fire Protection Association (U.S.A.):

Health: 2

Flammability: 3

Reactivity: 0

Specific hazard:

Protective Equipment:

Gloves. Lab coat. Vapor respirator. Be sure to use an approved/certified respirator or equivalent. Wear appropriate respirator when ventilation is inadequate. Splash goggles.

Section 16: Other Information

References: Not available.

Other Special Considerations: Not available.

Created: 10/10/2005 08:17 PM

Last Updated: 11/01/2010 12:00 PM

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MATERIAL SAFETY DATA SHEET
29 CFR 1910.1200 OSHA Hazard
Communication Rule Format
Chem-Tel 24 Hour Emergency # 1-800-255-3924

MINE SAFETY APPLIANCES COMPANY
P.O. Box 426
Pittsburgh, PA 15230
PHONE (412) 967-3000

This product contains isobutene, oxygen and nitrogen, substances subject to the Pennsylvania Worker and Community Right-To-Know Act.

PRODUCT IDENTITY

LABEL IDENTITY - MSA P/N 815704 Calibration Check Gas, 100 ppm isobutylene in air

CHEMICAL NAME - Isobutylene, Oxygen, Nitrogen Mixture

ADDITIONAL IDENTITIES - MSA P/N 815704 calibration gas, MSA P/N 815704 Squirt Gas

FORMULA - C_4H_8 in Air

APPLICABLE CHEMICAL CONTENTS

	ppm	TWA
Isobutene (CAS 115-11-7)	100	None
Air	Balance	None

NOTE: Gas under pressure, 155 PSIG at 70°F, Approx. 11 liters gas at atmospheric pressure.

PHYSICAL AND CHEMICAL PROPERTIES

APPEARANCE AND ODOR - Colorless gas, faint odor

BOILING POINT - N/A

SPECIFIC GRAVITY (H_2O = 1) - N/A

VAPOR PRESSURE - N/A

PERCENT VOLATILE BY VOLUME - N/A

VAPOR DENSITY (AIR = 1) - > 1

SOLUBILITY IN WATER - Isobutylene - Insoluble
Oxygen - 3.2 cm^3/100 ml (25°C)
Nitrogen - 2.3 cm^3/100 ml (0°C)

N/A - Not Applicable

PHYSICAL HAZARD INFORMATION

PHYSICAL HAZARD - Compressed Gas, 155 PSIG at 70°F

CONDITIONS OR MATERIALS TO AVOID - None

FLASH POINT - N/A LEL - N/A UEL - N/A

EXTINGUISHING MEDIA - This calibration gas mixture is not flammable.

SPECIAL FIRE FIGHTING PROCEDURES - See next item.

UNUSUAL FIRE AND EXPLOSION HAZARDS - Gas under pressure, 155 PSIG at 70°F. Do not exceed 120°F.

HEALTH HAZARDS

HEALTH HAZARDS - None Known for 100 ppm Isobutylene in Air. Isobutylene Inhalation Rat LC_{50}: 620 Gm/M^3/4H. Isobutylene Inhalation Mouse LC_{50}: 415 Gm/M^3/2H.

SIGNS AND SYMPTOMS OF EXPOSURE - N/A to this gas mixture.

PRIMARY ROUTES OF ENTRY - Inhalation

TARGET ORGANS - No information. Isobutylene is an asphyxiant and a weak anesthetic.

MEDICAL CONDITIONS GENERALLY RECOGNIZED AS BEING AGGRAVATED BY EXPOSURE - No information

EXPOSURE LIMITS - None (ACGIH 2005)

CARCINOGENICITY DATA - Component gases are not listed by NIOSH RTECS, OSHA, NTP or IARC.

EMERGENCY AND FIRST AID PROCEDURES - None

SAFE HANDLING AND USE

HYGIENIC PRACTICES - Avoid breathing gas

PROTECTIVE MEASURES DURING REPAIR AND MAINTENANCE OF CONTAMINATED EQUIPMENT - N/A

PROCEDURES FOR SPILL OR LEAK CLEANUP - Ventilate area

WASTE DISPOSAL - Do not puncture or incinerate cylinder. Before discarding cylinder, slowly release contents to a safe exhaust. Dispose of cylinder in accordance with local, state and federal regulations.

STORAGE - Store in a cool, dry, well-ventilated area. Do not exceed 120°F.

CONTROL MEASURES

PERSONAL PROTECTIVE EQUIPMENT - Due to the limited amount of gas in the cylinder, and the low release rate employed in instrument calibration, respiratory protection is not indicated under conditions of intended use.

ENGINEERING CONTROLS - None

WORK PRACTICES - Use in well-ventilated areas. Follow the calibration procedure detailed in the MSA instruction manual provided with the instrument under calibration.

DATE OF PREPARATION - Rev. 5, June 2005.

WARNING: This is a hazardous chemical product. By following the directions and warnings provided with this product, the hazards associated with the use of this product can be greatly reduced but never entirely eliminated. Mine Safety Appliances Company makes no warranties, expressed or implied, with respect to this product and EXPRESSLY DISCLAIMS THE WARRANTY OF MERCHANTABILITY AND ANY WARRANTY OF FITNESS FOR A PARTICULAR PURPOSE. Users assume all risks in handling, using or storing this product.

MATERIAL SAFETY DATA SHEET

ALCONOX®

Prepared to U.S. OSHA, CMA, ANSI, Canadian WHMIS, Australian WorkSafe, Japanese Industrial Standard JIS Z 7250:2000, and European Union REACH Regulations

SECTION 1 - PRODUCT AND COMPANY IDENTIFICATION

PRODUCT NAME:	**ALCONOX®**
CHEMICAL FAMILY NAME:	Detergent.
PRODUCT USE:	Critical-cleaning detergent for laboratory, healthcare and industrial applications
U.N. NUMBER:	Not Applicable
U.N. DANGEROUS GOODS CLASS:	Non-Regulated Material
SUPPLIER/MANUFACTURER'S NAME:	Alconox, Inc.
ADDRESS:	30 Glenn St., Suite 309, White Plains, NY 10603. USA
EMERGENCY PHONE:	**TOLL-FREE in USA/Canada** 800-255-3924
	International calls 813-248-0585
BUSINESS PHONE:	914-948-4040
DATE OF PREPARATION:	May 2011
DATE OF LAST REVISION:	February 2008

SECTION 2 - HAZARDS IDENTIFICATION

EMERGENCY OVERVIEW: This product is a white granular powder with little or no odor. Exposure can be irritating to eyes, respiratory system and skin. It is a non-flammable solid. The Environmental effects of this product have not been investigated.

US DOT SYMBOLS

Non-Regulated

CANADA (WHMIS) SYMBOLS

EUROPEAN and (GHS) Hazard Symbols

Signal Word: **Warning!**

EU LABELING AND CLASSIFICATION:

Classification of the substance or mixture according to Regulation (EC) No1272/2008 Annex 1
EC# 205-633-8 This substance is not classified in the Annex I of Directive 67/548/EEC
EC# 268-356-1 This substance is not classified in the Annex I of Directive 67/548/EEC
EC# 231-838-7 This substance is not classified in the Annex I of Directive 67/548/EEC
EC# 231-767-1 This substance is not classified in the Annex I of Directive 67/548/EEC
EC# 207-638-8 Index# 011-005-00-2
EC# 205-788-1 This substance is not classified in the Annex I of Directive 67/548/EEC

GHS Hazard Classification(s):

Eye Irritant Category 2A

Hazard Statement(s):

H319: Causes serious eye irritation

Precautionary Statement(s):

P260: Do not breath dust/fume/gas/mist/vapors/spray
P264: Wash hands thoroughly after handling
P271: Use only in well ventilated area.
P280: Wear protective gloves/protective clothing/eye protection/face protection/

Hazard Symbol(s):

[Xi] Irritant

Risk Phrases:
R20: Harmful by inhalation
R36/37/38: Irritating to eyes, respiratory system and skin

Safety Phrases:
S8: Keep container dry
S22: Do not breath dust
S24/25: Avoid contact with skin and eyes

HEALTH HAZARDS OR RISKS FROM EXPOSURE:

ACUTE: Exposure to this product may cause irritation of the eyes, respiratory system and skin. Ingestion may cause gastrointestinal irritation including pain, vomiting or diarrhea.

CHRONIC: This product contains an ingredient which may be corrosive.

TARGET ORGANS: ACUTE: Eye, respiratory System, Skin CHRONIC: None Known

SECTION 3 - COMPOSITION and INFORMATION ON INGREDIENTS

HAZARDOUS INGREDIENTS:	CAS #	EINECS #	ICSC #	WT %	HAZARD CLASSIFICATION; RISK PHRASES
Sodium Bicarbonate	144-55-8	205-633-8	1044	33 - 43%	HAZARD CLASSIFICATION: None RISK PHRASES: None
Sodium (C10 – C16) Alkylbenzene Sulfonate	68081-81-2	268-356-1	Not Listed	10 – 20%	HAZARD CLASSIFICATION: None RISK PHRASES: None
Sodium Tripolyphosphate	7758-29-4	231-838-7	1469	5 - 15%	HAZARD CLASSIFICATION: None RISK PHRASES: None
Tetrasodium Pyrophosphate	7722-88-5	231-767-1	1140	5 - 15%	HAZARD CLASSIFICATION: None RISK PHRASES: None
Sodium Carbonate	497-19-8	207-638-8	1135	1 - 10%	HAZARD CLASSIFICATION: [Xi] Irritant RISK PHRASES: R36
Sodium Alcohol Sulfate	151-21-3	205-788-1	0502	1 – 5%	HAZARD CLASSIFICATION: None RISK PHRASES: None
Balance of other ingredients are non-hazardous or less than 1% in concentration (or 0.1% for carcinogens, reproductive toxins, or respiratory sensitizers).					

NOTE: ALL WHMIS required information is included in appropriate sections based on the ANSI Z400.1-2004 format. This product has been classified in accordance with the hazard criteria of the CPR and the MSDS contains all the information required by the CPR, EU Directives and the Japanese Industrial Standard *JIS Z 7250: 2000*.

SECTION 4 - FIRST-AID MEASURES

Contaminated individuals of chemical exposure must be taken for medical attention if any adverse effect occurs. Rescuers should be taken for medical attention, if necessary. Take copy of label and MSDS to health professional with contaminated individual.

EYE CONTACT: If product enters the eyes, open eyes while under gentle running water for at least 15 minutes. Seek medical attention if irritation persists.

SKIN CONTACT: Wash skin thoroughly after handling. Seek medical attention if irritation develops and persists. Remove contaminated clothing. Launder before re-use.

INHALATION: If breathing becomes difficult, remove victim to fresh air. If necessary, use artificial respiration to support vital functions. Seek medical attention if breathing dificulty continues.

INGESTION: If product is swallowed, call physician or poison control center for most current information. If professional advice is not available, do not induce vomiting. Never induce vomiting or give diluents (milk or water) to someone who is unconscious, having convulsions, or who cannot swallow. Seek medical advice. Take a copy of the label and/or MSDS with the victim to the health professional.

MEDICAL CONDITIONS AGGRAVATED BY EXPOSURE: Pre-existing skin, or eye problems may be aggravated by prolonged contact.

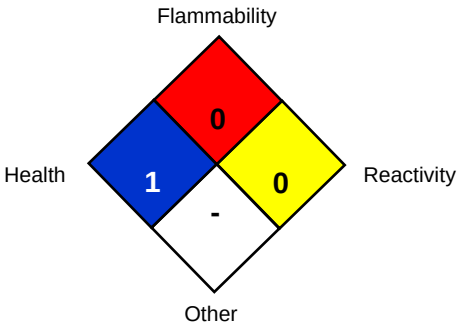
RECOMMENDATIONS TO PHYSICIANS: Treat symptoms and reduce over-exposure.

SECTION 5 - FIRE-FIGHTING MEASURES

FLASH POINT: Not Flammable
AUTOIGNITION TEMPERATURE: Not Applicable
FLAMMABLE LIMITS (in air by volume, %): Lower (LEL): NA Upper (UEL): NA
FIRE EXTINGUISHING MATERIALS: As appropriate for surrounding fire. Carbon dioxide, foam, dry chemical, halon, or water spray.

UNUSUAL FIRE AND EXPLOSION HAZARDS: This product is non-flammable and has no known explosion hazards.
Explosion Sensitivity to Mechanical Impact: Not Sensitive.
Explosion Sensitivity to Static Discharge: Not Sensitive
SPECIAL FIRE-FIGHTING PROCEDURES: Incipient fire responders should wear eye protection. Structural firefighters must wear Self-Contained Breathing Apparatus and full protective equipment. Isolate materials not yet involved in the fire and protect personnel. Move containers from fire area if this can be done without risk; otherwise, cool with carefully applied water spray. If possible, prevent runoff water from entering storm drains, bodies of water, or other environmentally sensitive areas.

NFPA RATING SYSTEM



HMIS RATING SYSTEM

HAZARDOUS MATERIAL IDENTIFICATION SYSTEM

HEALTH HAZARD (BLUE)	1
FLAMMABILITY HAZARD (RED)	0
PHYSICAL HAZARD (YELLOW)	0

PROTECTIVE EQUIPMENT			
EYES	RESPIRATORY	HANDS	BODY
	See Sect 8		See Sect 8

For Routine Industrial Use and Handling Applications

Hazard Scale: **0** = Minimal **1** = Slight **2** = Moderate **3** = Serious **4** = Severe * = Chronic hazard

SECTION 6 - ACCIDENTAL RELEASE MEASURES

SPILL AND LEAK RESPONSE: Personnel should be trained for spill response operations.
SPILLS: Contain spill if safe to do so. Prevent entry into drains, sewers, and other waterways. Sweep, shovel or vacuum spilled material and place in an appropriate container for re-use or disposal. Avoid dust generation if possible. Dispose of in accordance with applicable Federal, State, and local procedures (see Section 13, Disposal Considerations).

SECTION 7 - HANDLING and STORAGE

WORK PRACTICES AND HYGIENE PRACTICES: As with all chemicals, avoid getting this product ON YOU or IN YOU. Wash thoroughly after handling this product. Do not eat, drink, smoke, or apply cosmetics while handling this product. Avoid breathing dusts generated by this product. Use in a well-ventilated location. Remove contaminated clothing immediately.

STORAGE AND HANDLING PRACTICES: Containers of this product must be properly labeled. Store containers in a cool, dry location. Keep container tightly closed when not in use. Store away from strong acids or oxidizers.

SECTION 8 - EXPOSURE CONTROLS - PERSONAL PROTECTION

EXPOSURE LIMITS/GUIDELINES:

Chemical Name	CAS#	ACGIH TWA	OSHA TWA	SWA
Sodium Bicarbonate	144-55-8	10 mg/m³ Total Dust	15 mg/m³ Total Dust	10 mg/m³ Total Dust
Sodium (C10 – C16) Alkylbenzene Sulfonate	68081-81-2	10 mg/m³ Total Dust	15 mg/m³ Total Dust	10 mg/m³ Total Dust
Sodium Tripolyphosphate	7758-29-4	10 mg/m³ Total Dust	15 mg/m³ Total Dust	10 mg/m³ Total Dust
Tetrasodium Pyrophosphate	7722-88-5	5 mg/m³	5 mg/m³	5 mg/m³
Sodium Carbonate	497-19-8	10 mg/m³ Total Dust	15 mg/m³ Total Dust	10 mg/m³ Total Dust
Sodium Alcohol Sulfate	151-21-3	10 mg/m³ Total Dust	15 mg/m³ Total Dust	10 mg/m³ Total Dust

Currently, International exposure limits are not established for the components of this product. Please check with competent authority in each country for the most recent limits in place.

VENTILATION AND ENGINEERING CONTROLS: Use with adequate ventilation to ensure exposure levels are maintained below the limits provided below. Use local exhaust ventilation to control airborne dust. Ensure eyewash/safety shower stations are available near areas where this product is used.

The following information on appropriate Personal Protective Equipment is provided to assist employers in complying with OSHA regulations found in 29 CFR Subpart I (beginning at 1910.132) or equivalent standard of Canada, or standards of EU member states (including EN 149 for respiratory PPE, and EN 166 for face/eye protection), and those of Japan. Please reference applicable regulations and standards for relevant details.

RESPIRATORY PROTECTION: Based on test data, exposure limits should not be exceeded under normal use conditions when using Alconox Detergent. Maintain airborne contaminant concentrations below guidelines listed above, if applicable. If necessary, use only respiratory protection authorized in the U.S. Federal OSHA Respiratory Protection Standard (29 CFR 1910.134), equivalent U.S. State standards, Canadian CSA Standard Z94.4-93, the European Standard EN149, or EU member states.

EYE PROTECTION: Safety glasses. If necessary, refer to U.S. OSHA 29 CFR 1910.133 or appropriate Canadian Standards.

HAND PROTECTION: Use chemical resistant gloves to prevent skin contact.. If necessary, refer to U.S. OSHA 29 CFR 1910.138 or appropriate Standards of Canada.

BODY PROTECTION: Use body protection appropriate to prevent contact (e.g. lab coat, overalls). If necessary, refer to appropriate Standards of Canada, or appropriate Standards of the EU, Australian Standards, or relevant Japanese Standards.

SECTION 9 - PHYSICAL and CHEMICAL PROPERTIES

PHYSICAL STATE:	Solid
APPEARANCE & ODOR:	White granular powder with little or no odor.
ODOR THRESHOLD (PPM):	Not Available
VAPOR PRESSURE (mmHg):	Not Applicable
VAPOR DENSITY (AIR=1):	Not Applicable.
BY WEIGHT:	Not Available
EVAPORATION RATE (nBuAc = 1):	Not Applicable.
BOILING POINT (C°):	Not Applicable.
FREEZING POINT (C°):	Not Applicable.
pH:	9.5 (1% aqueous solution)
SPECIFIC GRAVITY 20°C: (WATER =1)	0.85 – 1.1
SOLUBILITY IN WATER (%)	>10% w/w
COEFFICIENT OF WATER/OIL DIST.:	Not Available
VOC:	None
CHEMICAL FAMILY:	Detergent

SECTION 10 - STABILITY and REACTIVITY

STABILITY: Product is stable
DECOMPOSITION PRODUCTS: When heated to decomposition this product produces Oxides of carbon (COx)
MATERIALS WITH WHICH SUBSTANCE IS INCOMPATIBLE: Strong acids and strong oxidizing agents.
HAZARDOUS POLYMERIZATION: Will not occur.
CONDITIONS TO AVOID: Contact with incompatible materials and dust generation.

SECTION 11 - TOXICOLOGICAL INFORMATION

TOXICITY DATA: Toxicity data is available for mixture:

CAS# 497-19-8 LD50 Oral (Rat)	4090 mg/kg
CAS# 497-19-8 LD50 Oral (Mouse)	6600 mg/kg
CAS# 497-19-8 LC50 Inhalation (Rat)	2300 mg/m³ 2H
CAS# 497-19-8 LC50 Inhalation (Mouse)	1200 mg/m³ 2H
CAS# 7758-29-4 LD50 Oral (Rat)	3120 mg/kg
CAS# 7758-29-4 LD50 Oral (Mouse)	3100 mg/kg
CAS# 7722-88-5 LD50 Oral (Rat)	4000 mg/kg

SUSPECTED CANCER AGENT: None of the ingredients are found on the following lists: FEDERAL OSHA Z LIST, NTP, CAL/OSHA, IARC and therefore is not considered to be, nor suspected to be a cancer-causing agent by these agencies.
IRRITANCY OF PRODUCT: Contact with this product can be irritating to exposed skin, eyes and respiratory system.
SENSITIZATION OF PRODUCT: This product is not considered a sensitizer.
REPRODUCTIVE TOXICITY INFORMATION: No information concerning the effects of this product and its components on the human reproductive system.

SECTION 12 - ECOLOGICAL INFORMATION

ALL WORK PRACTICES MUST BE AIMED AT ELIMINATING ENVIRONMENTAL CONTAMINATION.
ENVIRONMENTAL STABILITY: No Data available at this time.
EFFECT OF MATERIAL ON PLANTS or ANIMALS: No evidence is currently available on this product's effects on plants or animals.

EFFECT OF CHEMICAL ON AQUATIC LIFE: No evidence is currently available on this product's effects on aquatic life.

SECTION 13 - DISPOSAL CONSIDERATIONS

PREPARING WASTES FOR DISPOSAL: Waste disposal must be in accordance with appropriate Federal, State, and local regulations, those of Canada, Australia, EU Member States and Japan.

SECTION 14 - TRANSPORTATION INFORMATION

US DOT; IATA; IMO; ADR:

THIS PRODUCT IS NOT HAZARDOUS AS DEFINED BY 49 CFR 172.101 BY THE U.S. DEPARTMENT OF TRANSPORTATION.
PROPER SHIPPING NAME: Non-Regulated Material
HAZARD CLASS NUMBER and DESCRIPTION: Not Applicable
UN IDENTIFICATION NUMBER: Not Applicable
PACKING GROUP: Not Applicable.
DOT LABEL(S) REQUIRED: Not Applicable
NORTH AMERICAN EMERGENCY RESPONSE GUIDEBOOK NUMBER (2004): Not Applicable
MARINE POLLUTANT: None of the ingredients are classified by the DOT as a Marine Pollutant (as defined by 49 CFR 172.101, Appendix B)

U.S. DEPARTMENT OF TRANSPORTATION (DOT) SHIPPING REGULATIONS:
This product is not classified as dangerous goods, per U.S. DOT regulations, under 49 CFR 172.101.

TRANSPORT CANADA, TRANSPORTATION OF DANGEROUS GOODS REGULATIONS:
This product is not classified as Dangerous Goods, per regulations of Transport Canada.

INTERNATIONAL AIR TRANSPORT ASSOCIATION (IATA):
This product is not classified as Dangerous Goods, by rules of IATA:

INTERNATIONAL MARITIME ORGANIZATION (IMO) DESIGNATION:
This product is not classified as Dangerous Goods by the International Maritime Organization.

EUROPEAN AGREEMENT CONCERNING THE INTERNATIONAL CARRIAGE OF DANGEROUS GOODS BY ROAD (ADR):

This product is not classified by the United Nations Economic Commission for Europe to be dangerous goods.

SECTION 15 - REGULATORY INFORMATION

UNITED STATES REGULATIONS

SARA REPORTING REQUIREMENTS: This product is not subject to the reporting requirements of Sections 302, 304 and 313 of Title III of the Superfund Amendments and Reauthorization Act., as follows: None

TSCA: All components in this product are listed on the US Toxic Substances Control Act (TSCA) inventory of chemicals.

SARA 311/312:

Acute Health: Yes Chronic Health: No Fire: No Reactivity: No

U.S. SARA THRESHOLD PLANNING QUANTITY: There are no specific Threshold Planning Quantities for this product. The default Federal MSDS submission and inventory requirement filing threshold of 10,000 lb (4,540 kg) may apply, per 40 CFR 370.20.

U.S. CERCLA REPORTABLE QUANTITY (RQ): None

CALIFORNIA SAFE DRINKING WATER AND TOXIC ENFORCEMENT ACT (PROPOSITION 65): None of the ingredients are on the California Proposition 65 lists.

CANADIAN REGULATIONS:

CANADIAN DSL/NDSL INVENTORY STATUS: All of the components of this product are on the DSL Inventory

CANADIAN ENVIRONMENTAL PROTECTION ACT (CEPA) PRIORITIES SUBSTANCES LISTS: No component of this product is on the CEPA First Priorities Substance Lists.

CANADIAN WHMIS CLASSIFICATION and SYMBOLS: This product is categorized as a Controlled Product, Hazard Class D2B as per the Controlled Product Regulations

EUROPEAN ECONOMIC COMMUNITY INFORMATION:

EU LABELING AND CLASSIFICATION:

Classification of the mixture according to Regulation (EC) No1272/2008. See section 2 for details.

AUSTRALIAN INFORMATION FOR PRODUCT:

AUSTRALIAN INVENTORY OF CHEMICAL SUBSTANCES (AICS) STATUS: All components of this product are listed on the AICS.

STANDARD FOR THE UNIFORM SCHEDULING OF DRUGS AND POISONS: Not applicable.

JAPANESE INFORMATION FOR PRODUCT:

JAPANESE MINISTER OF INTERNATIONAL TRADE AND INDUSTRY (MITI) STATUS: The components of this product are not listed as Class I Specified Chemical Substances, Class II Specified Chemical Substances, or Designated Chemical Substances by the Japanese MITI.

INTERNATIONAL CHEMICAL INVENTORIES:

Listing of the components on individual country Chemical Inventories is as follows:

Asia-Pac:	Listed
Australian Inventory of Chemical Substances (AICS):	Listed
Korean Existing Chemicals List (ECL):	Listed
Japanese Existing National Inventory of Chemical Substances (ENCS):	Listed
Philippines Inventory if Chemicals and Chemical Substances (PICCS):	Listed
Swiss Giftliste List of Toxic Substances:	Listed
U.S. TSCA:	Listed

SECTION 16 - OTHER INFORMATION

PREPARED BY: Paul Eigbrett Global Safety Management, 10006 Cross Creek Blvd. Suite 440, Tampa, FL 33647

Disclaimer: To the best of Alconox, Inc. knowledge, the information contained herein is reliable and accurate as of this date; however, accuracy, suitability or completeness is not guaranteed and no warranties of any type either express or implied are provided. The information contained herein relates only to this specific product.

ANNEX:

IDENTIFIED USES OF ALCONOX® AND DIRECTIONS FOR USE

Used to clean: Healthcare instruments, laboratory ware, vacuum equipment, tissue culture ware, personal protective equipment, sampling apparatus, catheters, tubing, pipes, radioactive contaminated articles, optical parts, electronic components, pharmaceutical apparatus, cosmetics manufacturing equipment, metal castings, forgings and stampings, industrial parts, tanks and reactors. Authorized by USDA for use in federally inspected meat and poultry plants. Passes inhibitory residue test for water analysis. FDA certified.

Used to remove: Soil, grit, grime, buffing compound, slime, grease, oils, blood, tissue, salts, deposits, particulates, solvents, chemicals, radioisotopes, radioactive contaminations, silicon oils, mold release agents.

Surfaces cleaned: Corrosion inhibited formulation recommended for glass, metal, stainless steel, porcelain, ceramic, plastic, rubber and fiberglass. Can be used on soft metals such as copper, aluminum, zinc and magnesium if rinsed promptly. Corrosion testing may be advisable.

Cleaning method: Soak, brush, sponge, cloth, ultrasonic, flow through clean-inplace. Will foam—not for spray or machine use.

Directions: Make a fresh 1% solution (2 1/2 Tbsp. per gal., 1 1/4 oz. per gal. or 10 grams per liter) in cold, warm, or hot water. If available use warm water. Use cold water for blood stains. For difficult soils, raise water temperature and use more detergent. Clean by soak, circulate, wipe, or ultrasonic method. Not for spray machines, will foam. For nonabrasive scouring, make paste. Use 2% solution to soak frozen stopcocks. To remove silver tarnish, soak in 1% solution in aluminum container. RINSE THOROUGHLY—preferably with running water. For critical cleaning, do final or all rinsing in distilled, deionized, or purified water. For food contact surfaces, rinse with potable water. Used on a wide range of glass, ceramic, plastic, and metal surfaces. Corrosion testing may be advisable.

Material Safety Data Sheet
cis-1,2-Dichloroethylene, 97%

ACC# 97773

Section 1 - Chemical Product and Company Identification

MSDS Name: cis-1,2-Dichloroethylene, 97%
Catalog Numbers: AC113380000, AC113380025, AC113380100
Synonyms: cis-Acetylene dichloride.
Company Identification:

Acros Organics N.V.
One Reagent Lane
Fair Lawn, NJ 07410

For information in North America, call: 800-ACROS-01
For emergencies in the US, call CHEMTREC: 800-424-9300

Section 2 - Composition, Information on Ingredients

CAS#	Chemical Name	Percent	EINECS/ELINCS
156-59-2	cis-1,2-Dichloroethylene	97	205-859-7

Section 3 - Hazards Identification

EMERGENCY OVERVIEW

Appearance: Clear liquid. Flash Point: 6 deg C.
Warning! Flammable liquid and vapor. Harmful if inhaled. Unstabilized substance may polymerize. Causes eye and skin irritation. May be harmful if swallowed. May cause respiratory tract irritation.
Target Organs: Central nervous system, respiratory system, eyes, skin.

Potential Health Effects

Eye: Causes moderate eye irritation.
Skin: Causes moderate skin irritation. May cause dermatitis.
Ingestion: May cause gastrointestinal irritation with nausea, vomiting and diarrhea. May be harmful if swallowed. May cause central nervous system depression.
Inhalation: May cause respiratory tract irritation. May cause narcotic effects in high concentration. Eye irritation, vertigo, and nausea were reported in humans exposed at 2200 ppm.

Chronic: Not available. Some German investigators reported fatty degeneration of the liver upon repeated narcotic doses in rats and

Section 4 - First Aid Measures

Eyes: In case of contact, immediately flush eyes with plenty of water for a t least 15 minutes. Get medical aid.

Skin: In case of contact, flush skin with plenty of water. Remove contaminated clothing and shoes. Get medical aid if irritation develops and persists. Wash clothing before reuse.
Ingestion: If swallowed, do not induce vomiting unless directed to do so by medical personnel. Never give anything by mouth to an unconscious person. Get medical aid.
Inhalation: If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Get medical aid.
Notes to Physician: Treat symptomatically and supportively.

Section 5 - Fire Fighting Measures

General Information: As in any fire, wear a self-contained breathing apparatus in pressure-demand, MSHA/NIOSH (approved or equivalent), and full protective gear. Vapors may form an explosive mixture with air. Use water spray to keep fire-exposed containers cool. Flammable liquid and vapor. Fire or excessive heat may result in violent rupture of the container due to bulk polymerization. Vapors are heavier than air and may travel to a source of ignition and flash back. Vapors can spread along the ground and collect in low or confined areas. Hazardous polymerization may occur under fire conditions.
Extinguishing Media: Use water fog, dry chemical, carbon dioxide, or regular foam.
Flash Point: 6 deg C (42.80 deg F)
Autoignition Temperature: 440 deg C (824.00 deg F)
Explosion Limits, Lower:9.70 vol %
Upper: 12.80 vol %
NFPA Rating: (estimated) Health: 2; Flammability: 3; Instability: 2

Section 6 - Accidental Release Measures

General Information: Use proper personal protective equipment as indicated in Section 8.
Spills/Leaks: Absorb spill with inert material (e.g. vermiculite, sand or earth), then place in suitable container. Remove all sources of ignition. Use a spark-proof tool. Provide ventilation.

Section 7 - Handling and Storage

Handling: Wash thoroughly after handling. Remove contaminated clothing and wash before reuse. Ground and bond containers when transferring material. Use spark-proof tools and explosion proof equipment. Avoid contact with eyes, skin, and clothing. Empty containers retain product residue, (liquid and/or vapor), and can be dangerous. Avoid ingestion and inhalation. Do not pressurize, cut, weld, braze, solder, drill, grind, or expose empty containers to heat, sparks or open flames. Use only with adequate ventilation. Pure vapor will be uninhibited and may polymerize in vents or other confined spaces.
Storage: Keep away from sources of ignition. Store in a tightly closed container. Flammables-area. Store protected from light and air.

Section 8 - Exposure Controls, Personal Protection

Engineering Controls: Use process enclosure, local exhaust ventilation, or other engineering controls to control airborne levels below recommended exposure limits. Facilities storing or utilizing this material should be equipped with an eyewash facility and a safety shower.

Exposure Limits

Chemical Name	ACGIH	NIOSH	OSHA - Final PELs
cis-1,2-Dichloroethylene	200 ppm TWA	none listed	none listed

OSHA Vacated PELs: cis-1,2-Dichloroethylene: No OSHA Vacated PELs are listed for this chemical.

Personal Protective Equipment

Eyes: Wear chemical splash goggles.
Skin: Wear appropriate protective gloves to prevent skin exposure.
Clothing: Wear appropriate protective clothing to prevent skin exposure.
Respirators: Follow the OSHA respirator regulations found in 29 CFR 1910.134 or European Standard EN 149. Use a NIOSH/MSHA or European Standard EN 149 approved respirator if exposure limits are exceeded or if irritation or other symptoms are experienced.

Section 9 - Physical and Chemical Properties

Physical State: Liquid
Appearance: Clear
Odor: Pleasant odor
pH: Not available.
Vapor Pressure: 201 mm Hg @ 25 deg C
Vapor Density: 3.34 (air=1)
Evaporation Rate: Not available.
Viscosity: Not available.
Boiling Point: 60 deg C @ 760 mm Hg
Freezing/Melting Point:-80 deg C
Decomposition Temperature: Not available.
Solubility: Insoluble.
Specific Gravity/Density:1.2800
Molecular Formula:C2H2Cl2
Molecular Weight:96.94

Section 10 - Stability and Reactivity

Chemical Stability: Stable under normal temperatures and pressures. This material is a monomer and may polymerize under certain conditions if the stabilizer is lost.
Conditions to Avoid: Light, ignition sources, exposure to air, excess heat.
Incompatibilities with Other Materials: Strong oxidizing agents, strong bases, copper.
Hazardous Decomposition Products: Hydrogen chloride, phosgene, carbon monoxide, carbon dioxide.
Hazardous Polymerization: May occur.

Section 11 - Toxicological Information

RTECS#:
CAS# 156-59-2: KV9420000
LD50/LC50:
CAS# 156-59-2:
Inhalation, rat: LC50 = 13700 ppm;

.

Carcinogenicity:
CAS# 156-59-2: Not listed by ACGIH, IARC, NTP, or CA Prop 65.

Epidemiology: No data available.
Teratogenicity: No data available.
Reproductive Effects: No data available.
Mutagenicity: No data available.
Neurotoxicity: No data available.
Other Studies:

Section 12 - Ecological Information

No information available.

Section 13 - Disposal Considerations

Chemical waste generators must determine whether a discarded chemical is classified as a hazardous waste. US EPA guidelines for the classification determination are listed in 40 CFR Parts 261.3. Additionally, waste generators must consult state and local hazardous waste regulations to ensure complete and accurate classification.
RCRA P-Series: None listed.
RCRA U-Series: None listed.

Section 14 - Transport Information

	US DOT	Canada TDG
Shipping Name:	DOT regulated - small quantity provisions apply (see 49CFR173.4)	1,2-DICHLOROETHYLENE
Hazard Class:		3
UN Number:		UN1150
Packing Group:		II

Section 15 - Regulatory Information

US FEDERAL

TSCA
CAS# 156-59-2 is listed on the TSCA inventory.
Health & Safety Reporting List
None of the chemicals are on the Health & Safety Reporting List.
Chemical Test Rules
None of the chemicals in this product are under a Chemical Test Rule.
Section 12b
None of the chemicals are listed under TSCA Section 12b.
TSCA Significant New Use Rule
None of the chemicals in this material have a SNUR under TSCA.
CERCLA Hazardous Substances and corresponding RQs

None of the chemicals in this material have an RQ.

SARA Section 302 Extremely Hazardous Substances

None of the chemicals in this product have a TPQ.

Section 313 No chemicals are reportable under Section 313.

Clean Air Act:

This material does not contain any hazardous air pollutants.
This material does not contain any Class 1 Ozone depletors.
This material does not contain any Class 2 Ozone depletors.

Clean Water Act:

None of the chemicals in this product are listed as Hazardous Substances under the CWA.
None of the chemicals in this product are listed as Priority Pollutants under the CWA.
None of the chemicals in this product are listed as Toxic Pollutants under the CWA.

OSHA:

None of the chemicals in this product are considered highly hazardous by OSHA.

STATE

CAS# 156-59-2 can be found on the following state right to know lists: Pennsylvania, Massachusetts.

California Prop 65

California No Significant Risk Level: None of the chemicals in this product are listed.

European / International Regulations

European Labeling in Accordance with EC Directives

Hazard Symbols:

XN F

Risk Phrases:

R 11 Highly flammable.
R 20 Harmful by inhalation.
R 52/53 Harmful to aquatic organisms, may cause long-term adverse effects in the aquatic environment.

Safety Phrases:

S 16 Keep away from sources of ignition - No smoking.
S 29 Do not empty into drains.
S 7 Keep container tightly closed.
S 61 Avoid release to the environment. Refer to special instructions /safety data sheets.

WGK (Water Danger / Protection)

CAS# 156-59-2: No information available.

Canada - DSL / NDSL

CAS# 156-59-2 is listed on Canada's NDSL List.

Canada - WHMIS

WHMIS: Not available.

This product has been classified in accordance with the hazard criteria of the Controlled Products Regulations and the MSDS contains all of the information required by those regulations.

Canadian Ingredient Disclosure List

Section 16 - Additional Information

MSDS Creation Date: 2/09/1998
Revision #5 Date: 3/16/2007

The information above is believed to be accurate and represents the best information currently available to us.

However, we make no warranty of merchantability or any other warranty, express or implied, with respect to such information, and we assume no liability resulting from its use. Users should make their own investigations to determine the suitability of the information for their particular purposes. In no event shall Fisher be liable for any claims, losses, or damages of any third party or for lost profits or any special, indirect, incidental, consequential or exemplary damages, howsoever arising, even if Fisher has been advised of the possibility of such damages.

OAKTON® MATERIAL SAFETY DATA SHEET

Section 1. Chemical Product and Company Identification

Catalog Number(s)

00606-10, 00653-15, 00653-16, 00653-18, 00653-20, 00653-23, 00653-27, 00653-32, 00653-47, 00653-50, 00653-89, 35653-09, 35653-10, 35653-11, 35653-12, 35653-13, 35656-18, 35656-47

Product Identity

CONDUCTIVITY STANDARD SOLUTIONS, < 90 mS

Manufacturer's Name	Emergency Telephone Number (24 hr)
RICCA CHEMICAL COMPANY	CHEMTREC®: 800-424-9300
Address (Number, Street, City, State, and ZIP Code)	Telephone Number For Information
P.O. Box 13090	817-461-5601
	Date Prepared
Arlington, Texas 76094	3-17-2000

Section 2. Composition / Information on Ingredients

Component	CAS Registry #	Percent Concentration	Exposure Limits ACGIH TLV	Exposure Limits OSHA PEL
Potassium Chloride	7447-40-7	< 6	N/A	N/A
Water, Deionized	7732-18-5	Balance	N/A	N/A

Section 3. Hazards Identification

☆☆☆

INHALATION: Remove to fresh air. Give artificial respiration if necessary.

SKIN CONTACT: Flush with plenty of water for at least 15 minutes.

INGESTION: Dilute with water or milk. Call a physician if necessary.

Section 5. Fire Fighting Measures

FLAMMABLE PROPERTIES:

FLASH POINT: N/A METHOD USED: N/A

FLAMMABLE LIMITS

LFL: N/A UFL: N/A

EXTINGUISHING MEDIA: Use any means suitable for extinguishing surrounding fire.

FIRE & EXPLOSION HAZARDS: Not considered to be a fire or explosion hazard.

FIRE FIGHTING INSTRUCTIONS: Use normal procedures/instructions.

FIRE FIGHTING EQUIPMENT: Use protective clothing and breathing equipment appropriate for the surrounding fire.

Section 6. Accidental Release Measures

Absorb with suitable material (paper towels, etc.) and dispose of in accordance with local regulations. Small amounts may be flushed to the sewer with plenty of water.

Section 7. Handling and Storage

As with all chemicals, wash hands thoroughly after handling. Avoid contact with eyes and skin. Protect from freezing and physical damage. SAFETY STORAGE CODE: GENERAL

Section 8. Exposure Controls / Personal Protection

ENGINEERING CONTROLS: No specific controls are needed. Normal room ventilation is adequate.

RESPIRATORY PROTECTION: Normal room ventilation is adequate.

SKIN PROTECTION: Chemical resistant gloves are recommended.

EYE PROTECTION: Safety glasses or goggles.

Section 9. Physical and chemical Properties

APPEARANCE:	Clear, colorless liquid	**pH:**	approximately 7
ODOR:	Odorless	**BOILING POINT (^{o}C):**	approximately 100
SOLUBILITY IN WATER:	Infinite	**MELTING POINT (^{o}C):**	approximately 0
SPECIFIC GRAVITY:	approximately 1.0 – 1.04	**VAPOR PRESSURE:**	N/A

Section 10. Stability and Reactivity

CHEMICAL STABILITY: Stable under normal conditions of use and storage.

INCOMPATIBILITY: Bromine Trifluoride, Potassium Permanganate plus Sulfuric Acid.

HAZARDOUS DECOMPOSITION PRODUCTS: Oxides of Potassium.

HAZARDOUS POLYMERIZATION: Will not occur.

Section 11. Toxicological Information

LD_{50}, Oral, Rat: 2600 mg/kg (Potassium Chloride), details of toxic effects not reported other than lethal dose value. Irritation: eye, rabbit (500mg/24 hr mild).

Section 12. Ecological Information

ECOTOXICOLOGICAL INFORMATION: No information found.

CHEMICAL FATE INFORMATION: No information found.

Section 13. Disposal Considerations

Dilute with water and flush to sewer if local regulations allow. If not allowed, save for recovery or recycling in an approved waste disposal facility. Always dispose of in accordance with local, state and federal regulations.

Section 14. Transport Information (Not meant to be all inclusive)

D.O.T. SHIPPING NAME:	Not regulated
D.O.T. HAZARD CLASS:	None
U.N. / N.A. NUMBER:	None
PACKING GROUP:	None
D.O.T. LABEL:	None

Section 15. Regulatory Information (Not meant to be all inclusive - selected regulation represented)

OSHA STATUS: The above items either do not contain any specifically hazardous material or the potentially hazardous material is present in such low concentration that the items do not present any immediate threat to health and safety. These items do not meet the OSHA Hazard Communication Standard (29 CFR 1910.1200) definition of a hazardous material.

TSCA STATUS: All components of this solution are listed on the TSCA Inventory.

CERCLA REPORTABLE QUANTITY: Not reportable

SARA TITLE III:

SECTION 302 EXTREMELY HAZARDOUS SUBSTANCES: No

SECTION 311/312 HAZARDOUS CATEGORIES: No

SECTION 313 TOXIC CHEMICALS: No

RCRA STATUS: No

CALIFORNIA PROPOSITION 65: Not listed

Section 16. Other Information

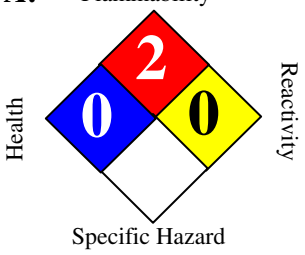
NFPA® Ratings:	**Health: 0**	**Flammability: 0**	**Reactivity: 0**	**Special Notice Key: None**
HMIS® Ratings:	**Health: 0**	**Flammability: 0**	**Reactivity: 0**	**Protective Equipment: B (Protective eyewear, gloves)**

Rev 1, 10-16-2000: (Section 1) added catalog numbers 35653-10, 35653-11, 35653-12, and 35653-13.
Rev 2, 12-06-2001: (Section 1) added catalog number 35653-09; revised description from 23µ - 80 mS.
Rev 3, 03-25-2003: Reviewed and approved, (Section 3) added mutation statement, (Section 11) added irritation data.
Rev 4, 03-20-2006: Reviewed and approved.

When handled properly by qualified personnel, the product described herein does not present a significant health or safety hazard. Alteration of its characteristics by concentration, evaporation, addition of other substances, or other means may present hazards not specifically addressed herein and which must be evaluated by the user. The information furnished herein is believed to be accurate and represents the best data currently available to us. No warranty, expressed or implied, is made and RICCA CHEMICAL COMPANY assumes no legal responsibility or liability whatsoever resulting from its use.

Material Safety Data Sheet
Diesel Low Sulfur (LSD) and Ultra Low Sulfur Diesel (ULSD)

NFPA:



TESORO

HMIS III:

HEALTH	1
FLAMMABILITY	2
PHYSICAL	0

0 = Insignificant, 1 = Slight, 2 = Moderate, 3 = High, 4 = Extreme

SECTION 1. PRODUCT AND COMPANY IDENTIFICATION

Product name	:	Diesel Low Sulfur (LSD) and Ultra Low Sulfur Diesel (ULSD)		
Synonyms	:	CARB Diesel, 888100004478		
MSDS Number	:	888100004478	**Version**	: 2.19
Product Use Description	:	Fuel		
Company	:	For: Tesoro Refining & Marketing Co. 19100 Ridgewood Parkway, San Antonio, TX 78259		
Tesoro Call Center	:	(877) 783-7676	**Chemtrec (Emergency Contact)**	: (800) 424-9300

SECTION 2. HAZARDS IDENTIFICATION

Emergency Overview

Regulatory status : This material is considered hazardous by the Occupational Safety and Health Administration (OSHA) Hazard Communication Standard (29 CFR 1910.1200).

Signal Word : WARNING

Hazard Summary : Toxic. Combustible Liquid

Potential Health Effects

Eyes : Eye irritation may result from contact with liquid, mists, and/or vapors.

Inhalation : Vapors or mists from this material can irritate the nose, throat, and lungs, and can cause signs and symptoms of central nervous system depression, depending on the concentration and duration of exposure.

Skin : Skin irritation leading to dermatitis may occur upon prolonged or repeated contact. Liquid may be absorbed through the skin in toxic amounts if large areas of skin are repeatedly exposed. Long-term, repeated skin contact may cause skin cancer

Ingestion : Harmful or fatal if swallowed. Do NOT induce vomiting. This material can irritate the mouth, throat, stomach, and cause nausea, vomiting, diarrhea and restlessness Aspiration hazard if liquid is inhaled into lungs, particularly from vomiting after ingestion. Aspiration may result in chemical pneumonia, severe lung damage, respiratory failure and even death.

Target Organs : Central nervous system, Eyes, Skin, Kidney, Liver

SECTION 3. COMPOSITION/INFORMATION ON INGREDIENTS

Component	CAS-No.	Weight %
Fuels, diesel, No 2; Gasoil - unspecified	68476-34-6	100%
Nonane	111-84-2	0 - 5%
Naphthalene	91-20-3	0 - 1%
1,2,4-Trimethylbenzene	95-63-6	0 - 2%
Xylene	1330-20-7	0 - 2%
Sulfur	7704-34-9	15 ppm maximum

SECTION 4. FIRST AID MEASURES

Inhalation : Move to fresh air. Give oxygen. If breathing is irregular or stopped, administer artificial respiration. Seek medical attention immediately.

Skin contact : Take off all contaminated clothing immediately. Wash off immediately with soap and plenty of water. Wash contaminated clothing before re-use. If skin irritation persists, seek medical attention immediately.

Eye contact : Remove contact lenses. Rinse thoroughly with plenty of water for at least 15 minutes. If symptoms persist, seek medical attention.

Ingestion : Do not induce vomiting without medical advice. If a person vomits when lying on his back, place him in the recovery position. Seek medical attention immediately.

Notes to physician : Symptoms: Dizziness, Discomfort, Headache, Nausea, Disorder, Vomiting, Lung edema, Aspiration may cause pulmonary edema and pneumonitis, Liver disorders, Kidney disorders.

SECTION 5. FIRE-FIGHTING MEASURES

Form : Liquid

Flash point : 38℃ Minimum for #1 Diesel, 52℃ Minimum for #2 Diesel

Auto Ignition temperature : 257 ℃ (495 ℉)

Lower explosive limit : 0.6 %(V)

Upper explosive limit : 4.7 %(V)

Suitable extinguishing media : Carbon dioxide (CO2), Water spray, Dry chemical, Foam, Keep containers and surroundings cool with water spray.

Specific hazards during fire fighting : Fire Hazard Do not use a solid water stream as it may scatter and spread fire. Cool closed containers exposed to fire with water spray.

Special protective equipment for fire-fighters	:	Wear self-contained breathing apparatus and protective suit. Use personal protective equipment.
Further information	:	Exposure to decomposition products may be a hazard to health. Isolate area around container involved in fire. Cool tanks, shells, and containers exposed to fire and excessive heat with water. For massive fires the use of unmanned hose holders or monitor nozzles may be advantageous to further minimize personnel exposure. Major fires may require withdrawal, allowing the tank to burn. Large storage tank fires typically require specially trained personnel and equipment to extinguish the fire, often including the need for properly applied fire fighting foam.

SECTION 6. ACCIDENTAL RELEASE MEASURES

Personal precautions	:	Evacuate nonessential personnel and remove or secure all ignition sources. Consider wind direction; stay upwind and uphill, if possible. Evaluate the direction of product travel, diking, sewers, etc. to contain spill areas. Spills may infiltrate subsurface soil and groundwater; professional assistance may be necessary to determine the extent of subsurface impact. Ensure adequate ventilation. Use personal protective equipment.
Environmental precautions	:	Carefully contain and stop the source of the spill, if safe to do so. Protect bodies of water by diking, absorbents, or absorbent boom, if possible. Do not flush down sewer or drainage systems, unless system is designed and permitted to handle such material. The use of fire fighting foam may be useful in certain situations to reduce vapors. The proper use of water spray may effectively disperse product vapors or the liquid itself, preventing contact with ignition sources or areas/equipment that require protection. Discharge into the environment must be avoided. If the product contaminates rivers and lakes or drains inform respective authorities.
Methods for cleaning up	:	Take up with sand or oil absorbing materials. Carefully shovel, scoop or sweep up into a waste container for reclamation or disposal - caution, flammable vapors may accumulate in closed containers. Response and clean-up crews must be properly trained and must utilize proper protective equipment (see Section 8).

SECTION 7. HANDLING AND STORAGE

Handling	:	Keep away from fire, sparks and heated surfaces. No smoking near areas where material is stored or handled. The product should only be stored and handled in areas with intrinsically safe electrical classification.
Advice on protection against fire and explosion	:	Hydrocarbon liquids including this product can act as a non-conductive flammable liquid (or static accumulators), and may form ignitable vapor-air mixtures in storage tanks or other containers. Precautions to prevent static-initated fire or explosion during transfer, storage or handling, include but are not limited to these examples: (1) Ground and bond containers during product transfers. Grounding and bonding may not be adequate protection to prevent ignition or explosion of hydrocarbon liquids and vapors that are static accumulators. (2) Special slow load procedures for "switch loading" must be followed to avoid the static ignition hazard that can exist when higher flash point material (such as fuel oil or diesel) is loaded into tanks previously containing low flash point products (such gasoline or naphtha). (3) Storage tank level floats must be effectively bonded. For more information on precautions to prevent static-initated fire or explosion, see NFPA 77, Recommended Practice on Static Electricity (2007), and API

Recommended Practice 2003, Protection Against Ignitions Arising Out of Static, Lightning, and Stray Currents (2008).

Dust explosion class : Not applicable

Requirements for storage areas and containers : Keep away from flame, sparks, excessive temperatures and open flame. Use approved containers. Keep containers closed and clearly labeled. Empty or partially full product containers or vessels may contain explosive vapors. Do not pressurize, cut, heat, weld or expose containers to sources of ignition. Store in a well-ventilated area. The storage area should comply with NFPA 30 "Flammable and Combustible Liquid Code". The cleaning of tanks previously containing this product should follow API Recommended Practice (RP) 2013 "Cleaning Mobile Tanks In Flammable and Combustible Liquid Service" and API RP 2015 "Cleaning Petroleum Storage Tanks".

Other data : Emergency eye wash capability should be available in the near proximity to operations presenting a potential splash exposure.

Advice on common storage Keep away from food, drink and animal feed. Incompatible with oxidizing agents. Incompatible with acids.

SECTION 8. EXPOSURE CONTROLS / PERSONAL PROTECTION

Exposure Guidelines

List	Components	CAS-No.	Type:	Value
OSHA Z1	Xylene	1330-20-7	PEL	100 ppm 435 mg/m3
	Naphthalene	91-20-3	PEL	10 ppm 50 mg/m3
ACGIH	Diesel Fuel	68476-30-2	TWA	100 mg/m3
	Xylene	1330-20-7	TWA	100 ppm
		1330-20-7	STEL	150 ppm
	Naphthalene	91-20-3	TWA	10 ppm
		91-20-3	STEL	15 ppm
	Nonane	111-84-2	TWA	200 ppm

Engineering measures : Use adequate ventilation to keep gas and vapor concentrations of this product below occupational exposure and flammability limits, particularly in confined spaces. Use only intrinsically safe electrical equipment approved for use in classified areas.

Eye protection : Safety glasses or goggles are recommended where there is a possibility of splashing or spraying.

Hand protection : Gloves constructed of nitrile, neoprene, or PVC are recommended. Consult manufacturer specifications for further information.

Skin and body protection : If needed to prevent skin contact, chemical protective clothing such as of DuPont

		TyChem®, Saranex or equivalent recommended based on degree of exposure. The resistance of specific material may vary from product to product as well as with degree of exposure.
Respiratory protection	:	A NIOSH/ MSHA-approved air-purifying respirator with organic vapor cartridges or canister may be permissible under certain circumstances where airborne concentrations are or may be expected to exceed exposure limits or for odor or irritation. Protection provided by air-purifying respirators is limited. Refer to OSHA 29 CFR 1910.134, ANSI Z88.2-1992, NIOSH Respirator Decision Logic, and the manufacturer for additional guidance on respiratory protection selection. Use a NIOSH/ MSHA-approved positive-pressure supplied-air respirator if there is a potential for uncontrolled release, exposure levels are not known, in oxygen-deficient atmospheres, or any other circumstance where an air-purifying respirator may not provide adequate protection.
Work / Hygiene practices	:	Emergency eye wash capability should be available in the near proximity to operations presenting a potential splash exposure. Use good personal hygiene practices. Avoid repeated and/or prolonged skin exposure. Wash hands before eating, drinking, smoking, or using toilet facilities. Do not use as a cleaning solvent on the skin. Do not use solvents or harsh abrasive skin cleaners for washing this product from exposed skin areas. Waterless hand cleaners are effective. Promptly remove contaminated clothing and launder before reuse. Use care when laundering to prevent the formation of flammable vapors which could ignite via washer or dryer. Consider the need to discard contaminated leather shoes and gloves.

SECTION 9. PHYSICAL AND CHEMICAL PROPERTIES

Form	:	Liquid
Appearance	:	Clear, straw colored
Odor	:	Characteristic petroleum (kerosene) odor
Flash point - typical	:	38°C Minimum for #1 Diesel, 52°C Minimum for #2 Diesel
Auto Ignition temperature	:	257 °C (495 °F)
Thermal decomposition	:	No decomposition if stored and applied as directed.
Lower explosive limit	:	0.6 %(V)
Upper explosive limit	:	4.7 %(V)
pH	:	Not applicable
Freezing point	:	No data available
Boiling point	:	154 - 372 °C(310° - 702 °F)
Vapor Pressure	:	< 2 mm Hg at 20 °C
Density	:	**0.86** g/cm3
Water solubility	:	Negligible
Viscosity, dynamic	:	1.7 - 40 mPa.s at 37.8 °C (100.0 °F)

Percent Volatiles	:	100 %
Conductivity (conductivity can be reduced by environmental factors such as a decrease in temperature		Diesel Fuel Oils at terminal load rack: At least 25 pS/m Ultra Low Sulfur Diesel (ULSD) without conductivity additive: 0 pS/m to 5 pS/m ULSD at terminal load rack with conductivity additive: At least 50 pS/m but conductivity may decrease from environmental factors such as temperature drop. JP-8 at terminal load rack: 150 pS/m to 600 pS/m

SECTION 10. STABILITY AND REACTIVITY

Conditions to avoid	:	Avoid high temperatures, open flames, sparks, welding, smoking and other ignition sources. Keep away from strong oxidizers. Viton ® ; Fluorel ®
Materials to avoid	:	Strong oxidizing agents. Peroxides
Hazardous decomposition products	:	Carbon monoxide, carbon dioxide and noncombusted hydrocarbons (smoke). Diesel exhaust particulates may be a lung hazard - see Section 11.
Thermal decomposition	:	No decomposition if stored and applied as directed.
Hazardous reactions	:	Keep away from oxidizing agents, and acidic or alkaline products.

SECTION 11. TOXICOLOGICAL INFORMATION

Carcinogenicity

NTP	:	Naphthalene (CAS-No.: 91-20-3)
IARC	:	Naphthalene (CAS-No.: 91-20-3)
OSHA	:	No component of this product which is present at levels greater than or equal to 0.1 % is identified as a carcinogen or potential carcinogen by OSHA.
CA Prop 65	:	WARNING! This product contains a chemical known to the State of California to cause cancer. naphthalene (CAS-No.: 91-20-3)
Skin irritation	:	Irritating to skin.
Eye irritation	:	Irritating to eyes.
Further information	:	Studies have shown that similar products produce skin cancer or skin tumors in laboratory animals following repeated applications without washing or removal. The significance of this finding to human exposure has not been determined. Other studies with active skin carcinogens have shown that washing the animal's skin with soap and water between applications reduced tumor formation. Positive mutagenicity results have been reported. Repeated over-exposure may cause liver and kidney injury IARC classifies whole diesel fuel exhaust particulates as probably carcinogenic to humans (Group 2A). NIOSH regards whole diesel fuel exhaust particulates as a potential cause of occupational lung cancer based on animal studies and limited evidence in humans.

Component:

Fuels, diesel, No 2; Gasoil - unspecified	68476-34-6	Acute oral toxicity: LD50 rat Dose: 5,001 mg/kg Acute dermal toxicity: LD50 rabbit

		Dose: 2,001 mg/kg Acute inhalation toxicity: LC50 rat Dose: 7.64 mg/l Exposure time: 4 h Skin irritation: Classification: Irritating to skin. Result: Severe skin irritation Eye irritation: Classification: Irritating to eyes. Result: Mild eye irritation
Nonane	111-84-2	Acute oral toxicity: LD50 mouse Dose: 218 mg/kg Acute inhalation toxicity: LC50 rat Exposure time: 4 h
Naphthalene	91-20-3	Acute oral toxicity: LD50 rat Dose: 2,001 mg/kg Acute dermal toxicity: LD50 rat Dose: 2,501 mg/kg Acute inhalation toxicity: LC50 rat Dose: 101 mg/l Exposure time: 4 h Skin irritation: Classification: Irritating to skin. Result: Mild skin irritation Eye irritation: Classification: Irritating to eyes. Result: Mild eye irritation Carcinogenicity: N11.00422130
1,2,4-Trimethylbenzene	95-63-6	Acute inhalation toxicity: LC50 rat Dose: 18 mg/l Exposure time: 4 h Skin irritation: Classification: Irritating to skin. Result: Skin irritation Eye irritation: Classification: Irritating to eyes. Result: Eye irritation
Xylene	1330-20-7	Acute oral toxicity: LD50 rat Dose: 2,840 mg/kg Acute dermal toxicity: LD50 rabbit Dose: ca. 4,500 mg/kg Acute inhalation toxicity: LC50 rat Dose: 6,350 mg/l Exposure time: 4 h Skin irritation: Classification: Irritating to skin. Result: Mild skin irritation Repeated or prolonged exposure may cause skin irritation and dermatitis, due to degreasing properties of the product. Eye irritation: Classification: Irritating to eyes. Result: Mild eye irritation

SECTION 12. ECOLOGICAL INFORMATION

Additional ecological : Keep out of sewers, drainage areas, and waterways. Report spills and releases, as

information applicable, under Federal and State regulations.

Component:

Naphthalene	91-20-3	Toxicity to algae: EC50 Species: Dose: 33 mg/l Exposure time: 24 h
1,2,4-Trimethylbenzene	95-63-6	Toxicity to fish: LC50 Species: Pimephales promelas (fathead minnow) Dose: 7.72 mg/l Exposure time: 96 h Acute and prolonged toxicity for aquatic invertebrates: EC50 Species: Daphnia Dose: 3.6 mg/l Exposure time: 48 h

SECTION 13. DISPOSAL CONSIDERATIONS

Disposal : In accordance with local and national regulations.

SECTION 14. TRANSPORT INFORMATION

CFR

Proper shipping name	: DIESEL FUEL
UN-No.	: UN1202 (NA 1993)
Class	: 3
Packing group	: III

TDG

Proper shipping name	: DIESEL FUEL
UN-No.	: UN1202 (NA 1993)
Class	: 3
Packing group	: III

IATA Cargo Transport

UN UN-No.	: UN1202 (NA 1993)
Description of the goods	: DIESEL FUEL
Class	: 3
Packaging group	: III
ICAO-Labels	: 3
Packing instruction (cargo aircraft)	: 366
Packing instruction (cargo aircraft)	: Y344

IATA Passenger Transport

UN UN-No.	: UN1202 (NA 1993)
Description of the goods	: DIESEL FUEL
Class	: 3
Packaging group	: III

	ICAO-Labels	: 3
	Packing instruction (passenger aircraft)	: 355
	Packing instruction (passenger aircraft)	: Y344
IMDG-Code		
	UN-No.	: UN 1202 (NA 1993)
	Description of the goods	: DIESEL FUEL
	Class	: 3
	Packaging group	: III
	IMDG-Labels	: 3
	EmS Number	: F-E S-E
	Marine pollutant	: No

SECTION 15. REGULATORY INFORMATION

OSHA Hazards : Combustible Liquid
Moderate skin irritant
Moderate eye irritant
Toxic by ingestion
POSSIBLE CANCER HAZARD

CERCLA SECTION 103 and SARA SECTION 304 (RELEASE TO THE ENVIROMENT)
The CERCLA definition of hazardous substances contains a "petroleum exclusion" clause which exempts crude oil. Fractions of crude oil, and products (both finished and intermediate) from the crude oil refining process and any indigenous components of such from the CERCLA Section 103 reporting requirements. However, other federal reporting requirements, including SARA Section 304, as well as the Clean Water Act may still apply.

TSCA Status : On TSCA Inventory

DSL Status : All components of this product are on the Canadian DSL list.

SARA 311/312 Hazards : Fire Hazard
Acute Health Hazard
Chronic Health Hazard

SARA III US. EPA Emergency Planning and Community Right-To-Know Act (EPCRA) SARA Title III Section 313 Toxic Chemicals (40 CFR 372.65) - Supplier Notification Required

Components	CAS-No.
Xylene	1330-20-7
1,2,4-Trimethylbenzene	95-63-6
Naphthalene	91-20-3

PENN RTK US. Pennsylvania Worker and Community Right-to-Know Law (34 Pa. Code Chap. 301-323)

Components	CAS-No.
Nonane	111-84-2

Naphthalene	91-20-3
1,2,4-Trimethylbenzene	95-63-6
xylene	1330-20-7
Fuels, diesel, No 2; Gasoil - unspecified	68476-34-6

MASS RTK — US. Massachusetts Commonwealth's Right-to-Know Law (Appendix A to 105 Code of Massachusetts Regulations Section 670.000)

Components	CAS-No.
Xylene	1330-20-7
1,2,4-Trimethylbenzene	95-63-6
Naphthalene	91-20-3
Nonane	111-84-2

NJ RTK — US. New Jersey Worker and Community Right-to-Know Act (New Jersey Statute Annotated Section 34:5A-5)

Components	CAS-No.
Nonane	111-84-2
Naphthalene	91-20-3
1,2,4-Trimethylbenzene	95-63-6
Xylene	1330-20-7
Fuels, diesel, No 2; Gasoil - unspecified	68476-34-6

California Prop. 65 : WARNING! This product contains a chemical known to the State of California to cause cancer.

Naphthalene 91-20-3

SECTION 16. OTHER INFORMATION

Further information

The information provided in this Safety Data Sheet is correct to the best of our knowledge, information and belief at the date of its publication. The information given is designed only as guidance for safe handling, use, processing, storage, transportation, disposal and release and is not to be considered a warranty or quality specification. The information relates only to the specific material designated and may not be valid for such material used in combination with any other materials or in any process, unless specified in the text.

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12/01/2011

1153, 1250, 1443, 1454, 1814, 1815, 1866, 1925

Material Safety Data Sheet

SECTION 1 PRODUCT AND COMPANY IDENTIFICATION

CHEVRON REGULAR UNLEADED GASOLINE

Product Number(s): CPS201000 [See Section 16 for Additional Product Numbers]
Synonyms: Calco Regular Unleaded Gasoline

Company Identification
Chevron Products Company
Marketing, MSDS Coordinator
6001 Bollinger Canyon Road
San Ramon, CA 94583
United States of America

Transportation Emergency Response
CHEMTREC: (800) 424-9300 or (703) 527-3887
Health Emergency
ChevronTexaco Emergency Information Center: Located in the USA. International collect calls accepted. (800) 231-0623 or (510) 231-0623
Product Information
Technical Information: (510) 242-5357

SPECIAL NOTES: This MSDS applies to: Federal Reformulated Gasoline, California Reformulated Gasoline, Wintertime Oxygenated Gasoline, Low RVP Gasoline and Conventional Gasoline.

SECTION 2 COMPOSITION/ INFORMATION ON INGREDIENTS

COMPONENTS	CAS NUMBER	AMOUNT
Gasoline	86290-81-5	100 %volume
Benzene	71-43-2	0.1 - 4.9 %volume
Ethyl benzene	100-41-4	0.1 - 3 %volume
Naphthalene	91-20-3	0.1 - 2 %volume
Ethanol	64-17-5	0 - 10 %volume
Methyl tert-butyl ether (MTBE)	1634-04-4	0 - 15 %volume
Tertiary amyl methyl ether (TAME)	994-05-8	0 - 17 %volume
Ethyl tert-butyl ether (ETBE)	637-92-3	0 - 18 %volume

Motor gasoline is considered a mixture by EPA under the Toxic Substances Control Act (TSCA). The refinery streams used to blend motor gasoline are all on the TSCA Chemical Substances Inventory. The appropriate CAS number for refinery blended motor gasoline is 86290-81-5. The product specifications of motor gasoline sold in your area will depend on applicable Federal and State regulations.

SECTION 3 HAZARDS IDENTIFICATION

****

EMERGENCY OVERVIEW

- EXTREMELY FLAMMABLE LIQUID AND VAPOR. VAPOR MAY CAUSE FLASH FIRE
- HARMFUL OR FATAL IF SWALLOWED - MAY CAUSE LUNG DAMAGE IF SWALLOWED
- VAPOR HARMFUL
- CAUSES SKIN IRRITATION
- CAUSES EYE IRRITATION
- LONG-TERM EXPOSURE TO VAPOR HAS CAUSED CANCER IN LABORATORY ANIMALS
- KEEP OUT OF REACH OF CHILDREN
- TOXIC TO AQUATIC ORGANISMS

****

IMMEDIATE HEALTH EFFECTS
Eye: Contact with the eyes causes irritation. Symptoms may include pain, tearing, reddening, swelling and impaired vision.
Skin: Contact with the skin causes irritation. Skin contact may cause drying or defatting of the skin. Symptoms may include pain, itching, discoloration, swelling, and blistering. Contact with the skin is not expected to cause an allergic skin response. Not expected to be harmful to internal organs if absorbed through the skin.
Ingestion: Because of its low viscosity, this material can directly enter the lungs, if swallowed, or if subsequently vomited. Once in the lungs it is very difficult to remove and can cause severe injury or death.
Inhalation: The vapor or fumes from this material may cause respiratory irritation. Symptoms of respiratory irritation may include coughing and difficulty breathing. Breathing this material at concentrations above the recommended exposure limits may cause central nervous system effects. Central nervous system effects may include headache, dizziness, nausea, vomiting, weakness, loss of coordination, blurred vision, drowsiness, confusion, or disorientation. At extreme exposures, central nervous system effects may include respiratory depression, tremors or convulsions, loss of consciousness, coma or death.

DELAYED OR OTHER HEALTH EFFECTS:
Reproduction and Birth Defects: This material is not expected to cause birth defects or other harm to the developing fetus based on animal data.
Cancer: Prolonged or repeated exposure to this material may cause cancer. Gasoline has been classified as a Group 2B carcinogen (possibly carcinogenic to humans) by the International Agency for Research on Cancer (IARC).

Contains benzene, which has been classified as a carcinogen by the National Toxicology Program (NTP) and a Group 1 carcinogen (carcinogenic to humans) by the International Agency for Research on Cancer (IARC).
Contains ethylbenzene which has been classified as a Group 2B carcinogen (possibly carcinogenic to humans) by the International Agency for Research on Cancer (IARC).
Contains naphthalene, which has been classified as a Group 2B carcinogen (possibly carcinogenic to humans) by the International Agency for Research on Cancer (IARC).

Whole gasoline exhaust has been classified as a Group 2B carcinogen (possibly carcinogenic to humans) by the International Agency for Research on Cancer (IARC).

Risk depends on duration and level of exposure. See Section 11 for additional information.

SECTION 4 FIRST AID MEASURES

Eye: Flush eyes with water immediately while holding the eyelids open. Remove contact lenses, if worn, after initial flushing, and continue flushing for at least 15 minutes. Get medical attention if irritation persists.
Skin: Wash skin with water immediately and remove contaminated clothing and shoes. Get medical attention if any symptoms develop. To remove the material from skin, use soap and water. Discard contaminated clothing and shoes or thoroughly clean before reuse.
Ingestion: If swallowed, get immediate medical attention. Do not induce vomiting. Never give anything by mouth to an unconscious person.
Inhalation: Move the exposed person to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Get medical attention if breathing difficulties continue.
Note to Physicians: Ingestion of this product or subsequent vomiting may result in aspiration of light hydrocarbon liquid, which may cause pneumonitis.

SECTION 5 FIRE FIGHTING MEASURES

See Section 7 for proper handling and storage.

FIRE CLASSIFICATION:
OSHA Classification (29 CFR 1910.1200): Flammable liquid.

NFPA RATINGS: Health: 1 Flammability: 3 Reactivity: 0

FLAMMABLE PROPERTIES:
Flashpoint: (Tagliabue Closed Cup) < -45 °C (< -49 °F)
Autoignition: > 280 °C (> 536 °F)
Flammability (Explosive) Limits (% by volume in air): Lower: 1.4 Upper: 7.6

EXTINGUISHING MEDIA: Dry Chemical, CO2, AFFF Foam or alcohol resistant foam if >15% volume polar solvents (oxygenates).

PROTECTION OF FIRE FIGHTERS:
Fire Fighting Instructions: Use water spray to cool fire-exposed containers and to protect personnel. For fires involving this material, do not enter any enclosed or confined fire space without proper protective equipment, including self-contained breathing apparatus.
Combustion Products: Highly dependent on combustion conditions. A complex mixture of airborne solids, liquids, and gases including carbon monoxide, carbon dioxide, and unidentified organic compounds will be evolved when this material undergoes combustion.

SECTION 6 ACCIDENTAL RELEASE MEASURES

Protective Measures: Eliminate all sources of ignition in the vicinity of the spill or released vapor. If this material is released into the work area, evacuate the area immediately. Monitor area with combustible gas indicator.
Spill Management: Stop the source of the release if you can do it without risk. Contain release to prevent further contamination of soil, surface water or groundwater. Clean up spill as soon as possible, observing precautions in Exposure Controls/Personal Protection. Use appropriate techniques such as applying non-combustible absorbent materials or pumping. All equipment used when handling the product must be grounded. A vapor suppressing foam may be used to reduce vapors. Use clean non-sparking tools to collect absorbed material. Where feasible and appropriate, remove contaminated soil. Place contaminated materials in disposable containers and dispose of in a manner consistent with applicable regulations.
Reporting: Report spills to local authorities and/or the U.S. Coast Guard's National Response Center at (800) 424-8802 as appropriate or required. This material is covered by EPA's Comprehensive Environmental Response, Compensation and Liability Act (CERCLA) Petroleum Exclusion. Therefore, releases to the environment may not be reportable under CERCLA.

SECTION 7 HANDLING AND STORAGE

Precautionary Measures: READ AND OBSERVE ALL PRECAUTIONS ON PRODUCT LABEL. This product presents an extreme fire hazard. Liquid very quickly evaporates, even at low temperatures, and forms vapor (fumes) which can catch fire and burn with explosive violence. Invisible vapor spreads easily and can be set on fire by many sources such as pilot lights, welding equipment, and electrical motors and switches. Never siphon gasoline by mouth.
Use only as a motor fuel. Do not use for cleaning, pressure appliance fuel, or any other such use. Do not store in open or unlabeled containers. Do not get in eyes, on skin, or on clothing. Do not taste or swallow. Do not breathe vapor or fumes. Wash thoroughly after handling. Keep out of the reach of children.
Unusual Handling Hazards: WARNING! Do not use as portable heater or appliance fuel. Toxic fumes may accumulate and cause death.
General Handling Information: Avoid contaminating soil or releasing this material into sewage and drainage systems and bodies of water.
Static Hazard: Electrostatic charge may accumulate and create a hazardous condition when handling this material. To minimize this hazard, bonding and grounding may be necessary but may not, by themselves, be sufficient. Review all operations which have the potential of generating an accumulation of electrostatic charge and/or a flammable atmosphere (including tank and container filling, splash filling, tank cleaning, sampling, gauging, switch loading, filtering, mixing, agitation, and vacuum truck operations) and use appropriate mitigating procedures. For more information, refer to OSHA Standard 29 CFR 1910.106, 'Flammable and Combustible Liquids', National Fire Protection Association (NFPA 77, 'Recommended Practice on Static Electricity', and/or the American Petroleum Institute (API) Recommended Practice 2003, 'Protection Against Ignitions Arising Out of Static, Lightning, and Stray Currents'. Improper filling of portable gasoline containers creates danger of fire. Only dispense gasoline into approved and properly labeled gasoline containers. Always place portable containers on the ground. Be sure pump nozzle is in contact with the container while filling. Do not use a nozzle's lock-open device. Do not fill portable containers that are inside a vehicle or truck/trailer bed.

General Storage Information: DO NOT USE OR STORE near heat, sparks or open flames. USE AND STORE ONLY IN WELL VENTILATED AREA. Keep container closed when not in use.
Container Warnings: Container is not designed to contain pressure. Do not use pressure to empty container or it may rupture with explosive force. Empty containers retain product residue (solid, liquid, and/or vapor) and can be dangerous. Do not pressurize, cut, weld, braze, solder, drill, grind, or expose such containers to heat, flame, sparks, static electricity, or other sources of ignition. They may explode and cause injury or death. Empty containers should be completely drained, properly closed, and promptly returned to a drum reconditioner or disposed of properly.

SECTION 8 EXPOSURE CONTROLS/PERSONAL PROTECTION

GENERAL CONSIDERATIONS:
Consider the potential hazards of this material (see Section 3), applicable exposure limits, job activities, and other substances in the work place when designing engineering controls and selecting personal protective equipment. If engineering controls or work practices are not adequate to

prevent exposure to harmful levels of this material, the personal protective equipment listed below is recommended. The user should read and understand all instructions and limitations supplied with the equipment since protection is usually provided for a limited time or under certain circumstances.

ENGINEERING CONTROLS:
Use process enclosures, local exhaust ventilation, or other engineering controls to control airborne levels below the recommended exposure limits.

PERSONAL PROTECTIVE EQUIPMENT
Eye/Face Protection: No special eye protection is normally required. Where splashing is possible, wear safety glasses with side shields as a good safety practice.
Skin Protection: No special protective clothing is normally required. Where splashing is possible, select protective clothing depending on operations conducted, physical requirements and other substances in the workplace. Suggested materials for protective gloves include: Chlorinated Polyethylene (or Chlorosulfonated Polyethylene), Nitrile Rubber, Polyurethane, Viton.
Respiratory Protection: Determine if airborne concentrations are below the recommended exposure limits. If not, wear an approved respirator that provides adequate protection from measured concentrations of this material, such as: Air-Purifying Respirator for Organic Vapors. When used as a fuel, this material can produce carbon monoxide in the exhaust. Determine if airborne concentrations are below the occupational exposure limit for carbon monoxide. If not, wear an approved positive-pressure air-supplying respirator.
Use a positive pressure air-supplying respirator in circumstances where air-purifying respirators may not provide adequate protection.

Occupational Exposure Limits:

Component	Limit	TWA	STEL	Ceiling	Notation
Benzene	ACGIH_TLV	.5 ppm	2.5 ppm		Skin A1
Benzene	OSHA_PEL	1 ppm	5 ppm		
Benzene	OSHA_Z2	10 ppm		25 ppm	
Ethanol	ACGIH_TLV	1000 ppm			A4
Ethanol	OSHA_PEL	1000 ppm			
Ethyl benzene	ACGIH_TLV	100 ppm	125 ppm		A3
Ethyl benzene	OSHA_PEL	100 ppm	125 ppm		
Ethyl tert-butyl ether (ETBE)	ACGIH_TLV	5 ppm			
Gasoline	ACGIH_TLV	300 ppm	500 ppm		A3
Gasoline	OSHA_PEL	300 ppm	500 ppm		
Methyl tert-butyl ether (MTBE)	ACGIH_TLV	50 ppm			A3
Naphthalene	ACGIH_TLV	10 ppm	15 ppm		Skin A4
Naphthalene	OSHA_PEL	10 ppm	15 ppm		
Tertiary amyl methyl ether (TAME)	CHEVRON		50 ppm		

Refer to the OSHA Benzene Standard (29 CFR 1910.1028) and Table Z-2 for detailed training, exposure monitoring, respiratory protection and medical surveillance requirements before using this product.

SECTION 9 PHYSICAL AND CHEMICAL PROPERTIES

Attention: the data below are typical values and do not constitute a specification.

Color: Colorless to yellow
Physical State: Liquid
Odor: Petroleum odor
pH: NA
Vapor Pressure: 5 psi - 15 psi (Typical) @ 37.8°C (100°F)
Vapor Density (Air = 1): 3 - 4 (Typical)
Boiling Point: 37.8°C (100°F) - 204.4°C (400°F) (Typical)
Solubility: Insoluble in water; miscible with most organic solvents.
Freezing Point: NA
Melting Point: NA
Specific Gravity: 0.7 g/ml - 0.8 g/ml @ 15.6°C (60.1°F)
Viscosity: <1 SUS @ 37.8°C (100°F)

SECTION 10 STABILITY AND REACTIVITY

Chemical Stability: This material is considered stable under normal ambient and anticipated storage and handling conditions of temperature and pressure.
Incompatibility With Other Materials: May react with strong oxidizing agents, such as chlorates, nitrates, peroxides, etc.
Hazardous Decomposition Products: None known (None expected)
Hazardous Polymerization: Hazardous polymerization will not occur.

SECTION 11 TOXICOLOGICAL INFORMATION

IMMEDIATE HEALTH EFFECTS
Eye Irritation: The Draize eye irritation mean score in rabbits for a 24-hour exposure was: 0/110.
Skin Irritation: For a 4-hour exposure, the Primary Irritation Index (PII) in rabbits is: 4.8/8.0.
Skin Sensitization: This material did not cause sensitization reactions in a Modified Buehler guinea pig test.
Acute Dermal Toxicity: 24 hour(s) LD50: >3.75g/kg (rabbit).
Acute Oral Toxicity: LD50: >5 ml/kg (rat)
Acute Inhalation Toxicity: 4 hour(s) LD50: >2000ppm (rat).

ADDITIONAL TOXICOLOGY INFORMATION:
Gasolines are highly volatile and can produce significant concentrations of vapor at ambient temperatures. Gasoline vapor is heavier than air and at high concentrations may accumulate in confined spaces to present both safety and heath hazards. When vapor exposures are low, or short duration and infrequent, such as during refuelling and tanker loading/unloading, neither total hydrocarbon nor components such as benzene are likely to result in any adverse health effects. In situations such as accidents or spills where exposure to gasoline vapor is potentially high, attention should be paid to potential toxic effects of specific components. Information about specific components in gasoline can be found in Sections 2, 8 and 15 of this MSDS. More detailed information on the health hazard of specific gasoline components can be obtained calling the Chevron Emergency Information Center (see Section 1 for phone numbers).

NEUROTOXICITY: Pathological misuse of solvents and gasoline, involving repeated and prolonged exposure to high concentrations of vapor is a significant exposure on which there are many reports in the medical literature. As with other solvents, persistent abuse involving repeated and prolonged exposures to high concentrations of vapor has been reported to result in central nervous system damage and eventually, death. In a study in which ten human volunteers were exposed for 30 minutes to approximately 200, 500 or 1000 ppm concentrations of gasoline vapor, irritation of the eyes was the only significant effect observed, based on both subjective and objective assessments. In an inhalation study, groups of 6 Fischer rats (3 male. 3 female) were exposed to 2056 ppm of wholy vaporized unleaded gasoline for 6 hours perday, 5 day per week for up to 18 months. Histopathology of the peripheral nervous system and spinal cord revealed no distal axonal neuropthy of the type associated with exposure to n-hexane even though gasoline contained 1.9% n-hexane. The authors concluded that gasoline treatment may have amplified the incidence and prominence of some naturally occurring age-related (subclinical) in the nervous system. BIRTH DEFECTS AND REPRODUCTIVE TOXICITY: An inhalation study with rats exposed to 0, 400 and 1600 ppm of wholly vaporized unleaded gasoline, 6 hours per day on day 6 through 16 of gestation, showed no teratogenic effects nor indication of toxicity to either the mother or the fetus. Another inhalation study in rats exposed to 3000, 6000, or 9000 ppm of gasoline vapor, 6 hours per day on day 6 through 20 of gestation, also showed no teratogenic effects nor indications of toxicity to either the mother or the fetus.

CHRONIC TOXICITY/CANCER: Wholly vaporized unleaded gasoline was used in a 3 month inhalation study. Groups of 40 rats (20 males, 20 female) and 8 squirrel monkeys (4 male, 4 female) were exposed 6 hours per day and 5 days per week for 13 weeks to 384 or 1552 ppm gasoline. One group of each species served as unexposed controls. The initial conclusion of this study was that inhalation of gasoline at airborne concentrations of up to 1522 ppm caused no toxicity in rats or monkeys. However, further histopathological examination of male rat kidneys on the highest dose group revealed an increased incidence and severity of regenerative epithelium and dilated tubules containing proteinaceous deposits. Lifetime inhalation of wholly vaporized unleaded gasoline at 2056 ppm has caused increased liver tumors in female mice. The mechanism of this response is still being investigated but it is thought to be an epigenetic process unique to the female mouse.

This exposure also caused kidney damage and eventually kidney cancer in male rats. No other animal model studied has shown these adverse

kidney effects and there is no physiological reason to believe that they would occur in man. EPA has concluded that mechanism by which wholly vaporzied unleaded gasoline causes kidney damage is unque to the male rat. The effects in that species (kidney damage and cancer) should not be used in human risk assesment. In their 1988 review of carcinogenic risk from gasoline, The International Agency for Research on Cancer (IARC) noted that, because published epidemiology studies did not include any exposure data, only occupations where gasoline exposure may have ocurred were reviewed. These included gasoline service station attendants and automobile mechanics. IARC also noted that there was no opportunity to separate effects of combustion products from those of gasoline itself. Although IARC allocated gasoline a final overall classification of Group 2B, i.e. possibly carcinogenic to humans, this was based on limited evidence in experimental animals plus supporting evidence including the presence in gasoline of benzene and 1, 3-butadiene. The actual evidence for carcinogenicity in humans was considered inadequate.

MUTAGENICITY: Gasoline was not mutagenic, with or without activation, in the Ames assay (Salmonella typhimurium), Saccharamyces cerevisesae, or mouse lymphoma assays. In addition, point mutations were not induced in human lymphocytes. Gasoline was not mutagenic when tested in the mouse dominant lethal assay. Administration of gasoline to rats did not cause chomosomal aberrations in their bone marrow cells. EPIDEMIOLOGY: To explore the health effects of workers potentially exposed to gasoline vapors in the marketing and distribution sectors of the petroleum industry, the American Petroleum Institute sponsored a cohort mortality study (Publication 4555), a nested case-control study (Publication 4551), and an exposure assessment study (Publication 4552). Histories of exposure to gasoline were reconstructed for cohort of more than 18,000 employees from four companies for the time period between 1946 and 1985. The results of the cohort mortality study indicated that there was no increased mortality from either kidney cancer or leukemia among marketing and marine distribution employees who were exposed to gasoline in the petroleum industry, when compared to the general population. More importantly, based on internal comparisons, there was no association between mortality from kidney cancer or leukemia and various indices of gasoline exposure. In particular, neither duration of employment, duration of exposure, age at first exposure, year of first exposure, job category, cumulative exposure, frequency of peak exposure, nor average intensity of exposure had any effect on kidney cancer or leukemia mortality. The results of the nested case-control study confirmed the findings of the original cohort study. That is, exposure to gasoline at the levels experienced by this cohort of distribution workers is not a significant risk factor for leukemia (all cell types), acute myeloid leukemia, kidney cancer or multiple myeloma.

SECTION 12 ECOLOGICAL INFORMATION

ECOTOXICITY

The 96 hour(s) LC50 for rainbow trout (Oncorhynchus mykiss) is 2.7 mg/l.
The 48 hour(s) LC50 for water flea (Daphnia magna) is 3.0 mg/l.
The 96 hour(s) LC50 for sheepshead minnow (Cyprinodon variegatus) is 8.3 mg/l.
The 96 hour(s) LC50 for mysid shrimp (Mysidopsis bahia) is 1.8 mg/l.
This material is expected to be toxic to aquatic organisms. Gasoline studies have been conducted in the laboratory under a variety of test conditions with a range of fish and invertebrate species. An even more extensive database is available on the aquatic toxicity of individual aromatic constituents. The majority of published studies do not identify the type of gasoline evaluated, or even provide distinguishing characteristics such as aromatic content or presence of lead alkyls. As a result, comparison of results among studies using open and closed vessels, different ages and species of test animals and different gasoline types, is difficult.

The bulk of the available literature on gasoline relates to the environmental impact of monoaromatic (BTEX) and diaromatic (naphthalene, methylnaphthalenes) constituents. In general, non-oxygenated gasoline exhibits some short-term toxicity to freshwater and marine organisms, especially under closed vessel or flow-through exposure conditions in the laboratory. The components which are the most prominent in the water soluble fraction and cause aquatic toxicity, are also highly volatile and can be readily biodegraded by microorganisms.

ENVIRONMENTAL FATE

This material is expected to be readily biodegradable. Following spillage, the more volatile components of gasoline will be rapidly lost, with concurrent dissolution of these and other constituents into the water. Factors such as local environmental conditions (temperature, wind, mixing or wave action, soil type, etc), photo-oxidation, biodegradation and adsorption onto suspended sediments, can contribute to the weathering of spilled gasoline.

The aqueous solubility of non-oxygenated unleaded gasoline, based on analysis of benzene, toluene, ethylbenzene+xylenes and naphthalene, is reported to be 112 mg/l. Solubility data on individual gasoline constituents also available.

SECTION 13 DISPOSAL CONSIDERATIONS

Use material for its intended purpose or recycle if possible. This material, if it must be discarded, may meet the criteria of a hazardous waste as defined by US EPA under RCRA (40 CFR 261) or other State and local regulations. Measurement of certain physical properties and analysis for regulated components may be necessary to make a correct determination. If this material is classified as a hazardous waste, federal law requires disposal at a licensed hazardous waste disposal facility.

SECTION 14 TRANSPORT INFORMATION

The description shown may not apply to all shipping situations. Consult 49CFR, or appropriate Dangerous Goods Regulations, for additional description requirements (e.g., technical name) and mode-specific or quantity-specific shipping requirements.

DOT Shipping Name: GASOLINE
DOT Hazard Class: 3 (Flammable Liquid)
DOT Identification Number: UN1203
DOT Packing Group: II

SECTION 15 REGULATORY INFORMATION

SARA 311/312 CATEGORIES:
1. Immediate (Acute) Health Effects: YES
2. Delayed (Chronic) Health Effects: YES
3. Fire Hazard: YES
4. Sudden Release of Pressure Hazard: NO
5. Reactivity Hazard: NO

REGULATORY LISTS SEARCHED:

4_I1=IARC Group 1	15=SARA Section 313
4_I2A=IARC Group 2A	16=CA Proposition 65
4_I2B=IARC Group 2B	17=MA RTK
05=NTP Carcinogen	18=NJ RTK
06=OSHA Carcinogen	19=DOT Marine Pollutant
09=TSCA 12(b)	20=PA RTK

The following components of this material are found on the regulatory lists indicated.

Benzene	15, 16, 17, 18, 20, 4_I1, 5, 6
Ethanol	17, 18, 20
Ethyl benzene	15, 17, 18, 20, 4_I2B
Gasoline	17, 18, 20
Methyl tert-butyl ether (MTBE)	15, 17, 18, 20, 9
Naphthalene	15, 16, 17, 18, 20, 4_I2B
Tertiary amyl methyl ether (TAME)	9

CERCLA REPORTABLE QUANTITIES(RQ)/SARA 302 THRESHOLD PLANNING QUANTITIES(TPQ):

Component	Component RQ	Component TPQ	Product RQ
Benzene	10 lbs	None	186 lbs
Ethanol	100 lbs	None	1961 lbs

Ethyl benzene	1000 lbs	None	34964 lbs
Methyl tert-butyl ether (MTBE)	1000 lbs	None	7513 lbs
Naphthalene	100 lbs	None	4000 lbs

CHEMICAL INVENTORIES:
CANADA: All the components of this material are on the Canadian DSL or have been notified under the New Substance Notification Regulations, but have not yet been published in the Canada Gazette.
UNITED STATES: All of the components of this material are on the Toxic Substances Control Act (TSCA) Chemical Inventory.

WHMIS CLASSIFICATION:
Class B, Division 2: Flammable Liquids
Class D, Division 2, Subdivision A: Very Toxic Material - Carcinogenicity
Class D, Division 2, Subdivision B: Toxic Material - Skin or Eye Irritation

SECTION 16 OTHER INFORMATION

NFPA RATINGS: Health: 1 Flammability: 3 Reactivity: 0

(0-Least, 1-Slight, 2-Moderate, 3-High, 4-Extreme, PPE:- Personal Protection Equipment Index recommendation, *- Chronic Effect Indicator). These values are obtained using the guidelines or published evaluations prepared by the National Fire Protection Association (NFPA) or the National Paint and Coating Association (for HMIS ratings).

Additional Product Number(s): CPS201023, CPS201054, CPS201055, CPS201075, CPS201090, CPS201105, CPS201106, CPS201120, CPS201121, CPS201122, CPS201126, CPS201128, CPS201131, CPS201136, CPS201141, CPS201142, CPS201148, CPS201153, CPS201158, CPS201161, CPS201162, CPS201168, CPS201181, CPS201185, CPS201186, CPS201188, CPS201216, CPS201217, CPS201218, CPS201236, CPS201237, CPS201238, CPS201266, CPS201267, CPS201268, CPS201277, CPS201278, CPS201279, CPS201286, CPS201287, CPS201289, CPS201296, CPS201297, CPS201298, CPS201849, CPS201850, CPS201855, CPS201856, CPS201857, CPS204000, CPS204001, CPS204002, CPS204003, CPS204010, CPS204011, CPS204022, CPS204023, CPS204046, CPS204047, CPS204070, CPS204071, CPS204088, CPS204089, CPS204104, CPS204105, CPS204116, CPS204117, CPS204140, CPS204141, CPS204164, CPS204165, CPS204188, CPS204189, CPS204200, CPS204201, CPS204212, CPS204213, CPS204224, CPS204225, CPS204248, CPS204249, CPS204272, CPS204273, CPS204290, CPS204291, CPS204322, CPS204323, CPS204324, CPS204350, CPS204352, CPS204354, CPS204356, CPS204358, CPS204359, CPS204364, CPS204365, CPS204370, CPS204371, CPS204376, CPS204377, CPS204382, CPS204383, CPS204388, CPS204389, CPS204394, CPS204395, CPS204400, CPS204401, CPS204406, CPS204407, CPS204412, CPS204413, CPS204418, CPS204419, CPS204424, CPS204425, CPS204430, CPS204431, CPS204436, CPS204437, CPS204442, CPS204446, CPS204450, CPS204454, CPS204458, CPS204462, CPS204466, CPS204467, CPS204484, CPS204485, CPS204502, CPS204503, CPS204520, CPS204521, CPS204538, CPS204539, CPS204556, CPS204557, CPS204574, CPS204575, CPS204592, CPS204593, CPS204610, CPS204611, CPS204628, CPS204629, CPS204646, CPS204647, CPS204664, CPS204665, CPS204682, CPS204690, CPS204691, CPS204696, CPS204697, CPS204702, CPS204703, CPS204708, CPS204709, CPS204721, CPS204722, CPS204727, CPS204728, CPS241765

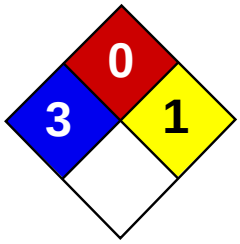
REVISION STATEMENT: This revision updates the following sections of this Material Safety Data Sheet: Section 1 (Product Codes). This Material Safety Data Sheet has been prepared using the ProSteward MSDS system.

ABBREVIATIONS THAT MAY HAVE BEEN USED IN THIS DOCUMENT:

TLV	-	Threshold Limit Value	TWA	-	Time Weighted Average
STEL	-	Short-term Exposure Limit	PEL	-	Permissible Exposure Limit
			CAS	-	Chemical Abstract Service Number
NDA	-	No Data Available	NA	-	Not Applicable
<=	-	Less Than or Equal To	>=	-	Greater Than or Equal To

Prepared according to the OSHA Hazard Communication Standard (29 CFR 1910.1200) and the ANSI MSDS Standard (Z400.1) by the ChevronTexaco Energy Research & Technology Company, 100 Chevron Way, Richmond, California 94802.

The above information is based on the data of which we are aware and is believed to be correct as of the date hereof. Since this information may be applied under conditions beyond our control and with which we may be unfamiliar and since data made available subsequent to the date hereof may suggest modifications of the information, we do not assume any responsibility for the results of its use. This information is furnished upon condition that the person receiving it shall make his own determination of the suitability of the material for his particular purpose.



Material Safety Data Sheet
Hydrochloric acid MSDS

Section 1: Chemical Product and Company Identification

Product Name: Hydrochloric acid

Catalog Codes: SLH1462, SLH3154

CAS#: Mixture.

RTECS: MW4025000

TSCA: TSCA 8(b) inventory: Hydrochloric acid

CI#: Not applicable.

Synonym: Hydrochloric Acid; Muriatic Acid

Chemical Name: Not applicable.

Chemical Formula: Not applicable.

Contact Information:

Sciencelab.com, Inc.
14025 Smith Rd.
Houston, Texas 77396

US Sales: **1-800-901-7247**
International Sales: **1-281-441-4400**

Order Online: ScienceLab.com

CHEMTREC (24HR Emergency Telephone), call:
1-800-424-9300

International CHEMTREC, call: 1-703-527-3887

For non-emergency assistance, call: 1-281-441-4400

Section 2: Composition and Information on Ingredients

Composition:

Name	CAS #	% by Weight
Hydrogen chloride	7647-01-0	20-38
Water	7732-18-5	62-80

Toxicological Data on Ingredients: Hydrogen chloride: GAS (LC50): Acute: 4701 ppm 0.5 hours [Rat].

Section 3: Hazards Identification

Potential Acute Health Effects:
Very hazardous in case of skin contact (corrosive, irritant, permeator), of eye contact (irritant, corrosive), of ingestion, . Slightly hazardous in case of inhalation (lung sensitizer). Non-corrosive for lungs. Liquid or spray mist may produce tissue damage particularly on mucous membranes of eyes, mouth and respiratory tract. Skin contact may produce burns. Inhalation of the spray mist may produce severe irritation of respiratory tract, characterized by coughing, choking, or shortness of breath. Severe over-exposure can result in death. Inflammation of the eye is characterized by redness, watering, and itching. Skin inflammation is characterized by itching, scaling, reddening, or, occasionally, blistering.

Potential Chronic Health Effects:
Slightly hazardous in case of skin contact (sensitizer). CARCINOGENIC EFFECTS: Classified 3 (Not classifiable for human.) by IARC [Hydrochloric acid]. MUTAGENIC EFFECTS: Not available. TERATOGENIC EFFECTS: Not available. DEVELOPMENTAL TOXICITY: Not available. The substance may be toxic to kidneys, liver, mucous membranes, upper respiratory tract, skin, eyes, Circulatory System, teeth. Repeated or prolonged exposure to the substance can produce target

organs damage. Repeated or prolonged contact with spray mist may produce chronic eye irritation and severe skin irritation. Repeated or prolonged exposure to spray mist may produce respiratory tract irritation leading to frequent attacks of bronchial infection. Repeated exposure to a highly toxic material may produce general deterioration of health by an accumulation in one or many human organs.

Section 4: First Aid Measures

Eye Contact:
Check for and remove any contact lenses. In case of contact, immediately flush eyes with plenty of water for at least 15 minutes. Cold water may be used. Get medical attention immediately.

Skin Contact:
In case of contact, immediately flush skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Cover the irritated skin with an emollient. Cold water may be used.Wash clothing before reuse. Thoroughly clean shoes before reuse. Get medical attention immediately.

Serious Skin Contact:
Wash with a disinfectant soap and cover the contaminated skin with an anti-bacterial cream. Seek immediate medical attention.

Inhalation:
If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Get medical attention immediately.

Serious Inhalation:
Evacuate the victim to a safe area as soon as possible. Loosen tight clothing such as a collar, tie, belt or waistband. If breathing is difficult, administer oxygen. If the victim is not breathing, perform mouth-to-mouth resuscitation. WARNING: It may be hazardous to the person providing aid to give mouth-to-mouth resuscitation when the inhaled material is toxic, infectious or corrosive. Seek immediate medical attention.

Ingestion:
If swallowed, do not induce vomiting unless directed to do so by medical personnel. Never give anything by mouth to an unconscious person. Loosen tight clothing such as a collar, tie, belt or waistband. Get medical attention immediately.

Serious Ingestion: Not available.

Section 5: Fire and Explosion Data

Flammability of the Product: Non-flammable.

Auto-Ignition Temperature: Not applicable.

Flash Points: Not applicable.

Flammable Limits: Not applicable.

Products of Combustion: Not available.

Fire Hazards in Presence of Various Substances: of metals

Explosion Hazards in Presence of Various Substances: Non-explosive in presence of open flames and sparks, of shocks.

Fire Fighting Media and Instructions: Not applicable.

Special Remarks on Fire Hazards:
Non combustible. Calcium carbide reacts with hydrogen chloride gas with incandescence. Uranium phosphide reacts with hydrochloric acid to release spontaneously flammable phosphine. Rubidium acetylene carbides burns with slightly warm hydrochloric acid. Lithium silicide in contact with hydrogen chloride becomes incandescent. When dilute hydrochloric acid is used, gas spontaneously flammable in air is evolved. Magnesium boride treated with concentrated hydrochloric acid produces spontaneously flammble gas. Cesium acetylene carbide burns hydrogen chloride gas. Cesium carbide ignites in contact with hydrochloric acid unless acid is dilute. Reacts with most metals to produce flammable Hydrodgen gas.

Special Remarks on Explosion Hazards:

Hydrogen chloride in contact with the following can cause an explosion, ignition on contact, or other violent/vigorous reaction: Acetic anhydride AgClO + CCl4 Alcohols + hydrogen cyanide, Aluminum Aluminum-titanium alloys (with HCl vapor), 2-Amino ethanol, Ammonium hydroxide, Calcium carbide Ca3P2 Chlorine + dinitroanilines (evolves gas), Chlorosulfonic acid Cesium carbide Cesium acetylene carbide, 1,1-Difluoroethylene Ethylene diamine Ethylene imine, Fluorine, HClO4 Hexalithium disilicide H2SO4 Metal acetylides or carbides, Magnesium boride, Mercuric sulfate, Oleum, Potassium permanganate, beta-Propiolactone Propylene oxide Rubidium carbide, Rubidium, acetylene carbide Sodium (with aqueous HCl), Sodium hydroxide Sodium tetraselenium, Sulfonic acid, Tetraselenium tetranitride, U3P4 , Vinyl acetate. Silver perchlorate with carbon tetrachloride in the presence of hydrochloric acid produces trichloromethyl perchlorate which detonates at 40 deg. C.

Section 6: Accidental Release Measures

Small Spill:
Dilute with water and mop up, or absorb with an inert dry material and place in an appropriate waste disposal container. If necessary: Neutralize the residue with a dilute solution of sodium carbonate.

Large Spill:
Corrosive liquid. Poisonous liquid. Stop leak if without risk. Absorb with DRY earth, sand or other non-combustible material. Do not get water inside container. Do not touch spilled material. Use water spray curtain to divert vapor drift. Use water spray to reduce vapors. Prevent entry into sewers, basements or confined areas; dike if needed. Call for assistance on disposal. Neutralize the residue with a dilute solution of sodium carbonate. Be careful that the product is not present at a concentration level above TLV. Check TLV on the MSDS and with local authorities.

Section 7: Handling and Storage

Precautions:
Keep locked up.. Keep container dry. Do not ingest. Do not breathe gas/fumes/ vapor/spray. Never add water to this product. In case of insufficient ventilation, wear suitable respiratory equipment. If ingested, seek medical advice immediately and show the container or the label. Avoid contact with skin and eyes. Keep away from incompatibles such as oxidizing agents, organic materials, metals, alkalis, moisture. May corrode metallic surfaces. Store in a metallic or coated fiberboard drum using a strong polyethylene inner package.

Storage: Keep container tightly closed. Keep container in a cool, well-ventilated area.

Section 8: Exposure Controls/Personal Protection

Engineering Controls:
Provide exhaust ventilation or other engineering controls to keep the airborne concentrations of vapors below their respective threshold limit value. Ensure that eyewash stations and safety showers are proximal to the work-station location.

Personal Protection:
Face shield. Full suit. Vapor respirator. Be sure to use an approved/certified respirator or equivalent. Gloves. Boots.

Personal Protection in Case of a Large Spill:
Splash goggles. Full suit. Vapor respirator. Boots. Gloves. A self contained breathing apparatus should be used to avoid inhalation of the product. Suggested protective clothing might not be sufficient; consult a specialist BEFORE handling this product.

Exposure Limits:
CEIL: 5 (ppm) from OSHA (PEL) [United States] CEIL: 7 (mg/m3) from OSHA (PEL) [United States] CEIL: 5 from NIOSH CEIL: 7 (mg/m3) from NIOSH TWA: 1 STEL: 5 (ppm) [United Kingdom (UK)] TWA: 2 STEL: 8 (mg/m3) [United Kingdom (UK)]Consult local authorities for acceptable exposure limits.

Section 9: Physical and Chemical Properties

Physical state and appearance: Liquid.

Odor: Pungent. Irritating (Strong.)

Taste: Not available.

Molecular Weight: Not applicable.

Color: Colorless to light yellow.

pH (1% soln/water): Acidic.

Boiling Point:
108.58 C @ 760 mm Hg (for 20.22% HCl in water) 83 C @ 760 mm Hg (for 31% HCl in water) 50.5 C (for 37% HCl in water)

Melting Point:
-62.25°C (-80°F) (20.69% HCl in water) -46.2 C (31.24% HCl in water) -25.4 C (39.17% HCl in water)

Critical Temperature: Not available.

Specific Gravity:
1.1- 1.19 (Water = 1) 1.10 (20%and 22% HCl solutions) 1.12 (24% HCl solution) 1.15 (29.57% HCl solution) 1.16 (32% HCl solution) 1.19 (37% and 38%HCl solutions)

Vapor Pressure: 16 kPa (@ 20°C) average

Vapor Density: 1.267 (Air = 1)

Volatility: Not available.

Odor Threshold: 0.25 to 10 ppm

Water/Oil Dist. Coeff.: Not available.

Ionicity (in Water): Not available.

Dispersion Properties: See solubility in water, diethyl ether.

Solubility: Soluble in cold water, hot water, diethyl ether.

Section 10: Stability and Reactivity Data

Stability: The product is stable.

Instability Temperature: Not available.

Conditions of Instability: Incompatible materials, water

Incompatibility with various substances:
Highly reactive with metals. Reactive with oxidizing agents, organic materials, alkalis, water.

Corrosivity:
Extremely corrosive in presence of aluminum, of copper, of stainless steel(304), of stainless steel(316). Non-corrosive in presence of glass.

Special Remarks on Reactivity:
Reacts with water especially when water is added to the product. Absorption of gaseous hydrogen chloride on mercuric sulfate becomes violent @ 125 deg. C. Sodium reacts very violently with gaseous hydrogen chloride. Calcium phosphide and hydrochloric acid undergo very energetic reaction. It reacts with oxidizers releasing chlorine gas. Incompatible with, alkali metals, carbides, borides, metal oxides, vinyl acetate, acetylides, sulphides, phosphides, cyanides, carbonates. Reacts with most metals to produce flammable Hydrogen gas. Reacts violently (moderate reaction with heat of evolution) with water especially when water is added to the product. Isolate hydrogen chloride from heat, direct sunlight, alkalies (reacts vigorously), organic materials, and oxidizers (especially nitric acid and chlorates), amines, metals, copper and alloys (e.g. brass), hydroxides, zinc (galvanized materials), lithium silicide (incandescence), sulfuric acid(increase in temperature and pressure) Hydrogen chloride gas is emitted when this product is in contact with sulfuric acid. Adsorption of Hydrochloric Acid onto silicon dioxide results in exothmeric reaction. Hydrogen chloride causes aldehydes and epoxides to violently polymerize. Hydrogen chloride or Hydrochloric Acid in contact with the folloiwng can cause explosion or ignition on contact or

Special Remarks on Corrosivity:

Highly corrosive. Incompatible with copper and copper alloys. It attacks nearly all metals (mercury, gold, platinium, tantalum, silver, and certain alloys are exceptions). It is one of the most corrosive of the nonoxidizing acids in contact with copper alloys. No corrosivity data on zinc, steel. Severe Corrosive effect on brass and bronze

Polymerization: Will not occur.

Section 11: Toxicological Information

Routes of Entry: Absorbed through skin. Dermal contact. Eye contact. Inhalation.

Toxicity to Animals:
Acute oral toxicity (LD50): 900 mg/kg [Rabbit]. Acute toxicity of the vapor (LC50): 1108 ppm, 1 hours [Mouse]. Acute toxicity of the vapor (LC50): 3124 ppm, 1 hours [Rat].

Chronic Effects on Humans:
CARCINOGENIC EFFECTS: Classified 3 (Not classifiable for human.) by IARC [Hydrochloric acid]. May cause damage to the following organs: kidneys, liver, mucous membranes, upper respiratory tract, skin, eyes, Circulatory System, teeth.

Other Toxic Effects on Humans:
Very hazardous in case of skin contact (corrosive, irritant, permeator), of ingestion, . Hazardous in case of eye contact (corrosive), of inhalation (lung corrosive).

Special Remarks on Toxicity to Animals:
Lowest Published Lethal Doses (LDL/LCL) LDL [Man] -Route: Oral; 2857 ug/kg LCL [Human] - Route: Inhalation; Dose: 1300 ppm/30M LCL [Rabbit] - Route: Inhalation; Dose: 4413 ppm/30M

Special Remarks on Chronic Effects on Humans:
May cause adverse reproductive effects (fetoxicity). May affect genetic material.

Special Remarks on other Toxic Effects on Humans:
Acute Potential Health Effects: Skin: Corrosive. Causes severe skin irritation and burns. Eyes: Corrosive. Causes severe eye irritation/conjuntivitis, burns, corneal necrosis. Inhalation: May be fatal if inhaled. Material is extremely destructive to tissue of the mucous membranes and upper respiratory tract. Inhalation of hydrochloric acid fumes produces nose, throat, and larryngeal burning, and irritation, pain and inflammation, coughing, sneezing, choking sensation, hoarseness, laryngeal spasms, upper respiratory tract edema, chest pains, as well has headache, and palpitations. Inhalation of high concentrations can result in corrosive burns, necrosis of bronchial epithelium, constriction of the larynx and bronchi, nasospetal perforation, glottal closure, occur, particularly if exposure is prolonged. May affect the liver. Ingestion: May be fatal if swallowed. Causes irritation and burning, ulceration, or perforation of the gastrointestinal tract and resultant peritonitis, gastric hemorrhage and infection. Can also cause nausea, vomitting (with "coffee ground" emesis), diarrhea, thirst, difficulty swallowing, salivation, chills, fever, uneasiness, shock, strictures and stenosis (esophogeal, gastric, pyloric). May affect behavior (excitement), the cardiovascular system (weak rapid pulse, tachycardia), respiration (shallow respiration), and urinary system (kidneys- renal failure, nephritis). Acute exposure via inhalation or ingestion can also cause erosion of tooth enamel. Chronic Potential Health Effects: dyspnea, bronchitis. Chemical pneumonitis and pulmonary edema can also

Section 12: Ecological Information

Ecotoxicity: Not available.

BOD5 and COD: Not available.

Products of Biodegradation:
Possibly hazardous short term degradation products are not likely. However, long term degradation products may arise.

Toxicity of the Products of Biodegradation: The products of degradation are less toxic than the product itself.

Special Remarks on the Products of Biodegradation: Not available.

Section 13: Disposal Considerations

Waste Disposal:

Waste must be disposed of in accordance with federal, state and local environmental control regulations.

Section 14: Transport Information

DOT Classification: Class 8: Corrosive material

Identification: : Hydrochloric acid, solution UNNA: 1789 PG: II

Special Provisions for Transport: Not available.

Section 15: Other Regulatory Information

Federal and State Regulations:
Connecticut hazardous material survey.: Hydrochloric acid Illinois toxic substances disclosure to employee act: Hydrochloric acid Illinois chemical safety act: Hydrochloric acid New York release reporting list: Hydrochloric acid Rhode Island RTK hazardous substances: Hydrochloric acid Pennsylvania RTK: Hydrochloric acid Minnesota: Hydrochloric acid Massachusetts RTK: Hydrochloric acid Massachusetts spill list: Hydrochloric acid New Jersey: Hydrochloric acid New Jersey spill list: Hydrochloric acid Louisiana RTK reporting list: Hydrochloric acid Louisiana spill reporting: Hydrochloric acid California Director's List of Hazardous Substances: Hydrochloric acid TSCA 8(b) inventory: Hydrochloric acid TSCA 4(a) proposed test rules: Hydrochloric acid SARA 302/304/311/312 extremely hazardous substances: Hydrochloric acid SARA 313 toxic chemical notification and release reporting: Hydrochloric acid CERCLA: Hazardous substances.: Hydrochloric acid: 5000 lbs. (2268 kg)

Other Regulations:
OSHA: Hazardous by definition of Hazard Communication Standard (29 CFR 1910.1200). EINECS: This product is on the European Inventory of Existing Commercial Chemical Substances.

Other Classifications:

WHMIS (Canada):
CLASS D-2A: Material causing other toxic effects (VERY TOXIC). CLASS E: Corrosive liquid.

DSCL (EEC):
R34- Causes burns. R37- Irritating to respiratory system. S26- In case of contact with eyes, rinse immediately with plenty of water and seek medical advice. S45- In case of accident or if you feel unwell, seek medical advice immediately (show the label where possible).

HMIS (U.S.A.):

Health Hazard: 3

Fire Hazard: 0

Reactivity: 1

Personal Protection:

National Fire Protection Association (U.S.A.):

Health: 3

Flammability: 0

Reactivity: 1

Specific hazard:

Protective Equipment:
Gloves. Full suit. Vapor respirator. Be sure to use an approved/certified respirator or equivalent. Wear appropriate respirator when ventilation is inadequate. Face shield.

Section 16: Other Information

References:
-Hawley, G.G.. The Condensed Chemical Dictionary, 11e ed., New York N.Y., Van Nostrand Reinold, 1987. -SAX, N.I. Dangerous Properties of Indutrial Materials. Toronto, Van Nostrand Reinold, 6e ed. 1984. -The Sigma-Aldrich Library of Chemical Safety Data, Edition II. -Guide de la loi et du règlement sur le transport des marchandises dangeureuses au canada. Centre de conformité internatinal Ltée. 1986.

Other Special Considerations: Not available.

Created: 10/09/2005 05:45 PM

Last Updated: 11/01/2010 12:00 PM

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MATERIAL SAFETY DATA SHEET

This Material Safety Data Sheet (MSDS) complies with the United Nations Globally Harmonized System (GHS) of Classification and Labeling, Second revised Edition and meets or exceeds the Canadian and United States Requirements for Hazard Communication

1. Product and Supplier Identification

Product Name: Methanol
GHS Product Identifier: Methanol

Recommended Use: Solvent, fuel, feedstock

Restrictions on Use: Do not use in a confined area without proper ventilation. Contact lenses may cause further damage in case of splash into eye. Avoid use near heat, flames, sparks, and other sources of ignition.

Product:	**Methanol (CH_3OH)**	**Methanex Tel. #:** (604) 661-2600
Synonyms:	Methanol, methyl hydrate, wood spirit, methyl hydroxide	**Emergency Tel. #:** 1-800-262-8200 **(CHEMTREC)** (Canada and USA)
Company Identification:	**Methanex Corporation 1800 Waterfront Centre 200 Burrard Street Vancouver, B.C. V6C 3M1**	
Importer:	**Methanex Methanol Company 15301 Dallas Parkway, Suite 900 Addison, Texas 75001 Telephone: (972) 702-0909**	

2. Hazards Identification

Classification: Flammable Liquid, Category 1, Acute Toxicity Category 1*, Reproductive Toxicity 1B, Specific Target Organ Toxicity (Repeated Exposure)

Label:

Hazard Communication: DANGER! Extremely flammable liquid and vapour. Fatal if swallowed.
May damage fertility or the unborn child (fetotoxic and teratogenic effects).
May cause damage to eyes and central nervous system if ingested or inhaled.

***Note:** Assigned to classification based on human experience rather than the strict application of classification criteria set out in the Recommendations on the Transport of Dangerous Goods, Model Regulations Special Provision 279.

Hazards: Colourless liquid, with a mild, characteristic alcohol odour when pure. Crude methanol may have a repulsive, pungent odour. Hygroscopic (moisture absorbing).

***FLAMMABLE LIQUID AND VAPOUR*:** Burns with a clean, clear flame, which is almost invisible in daylight, or a light blue flame. Can decompose at high temperatures forming carbon monoxide and formaldehyde.

Confined space toxicity hazard. Mild central nervous system depressant following inhalation, skin absorption or ingestion. May cause headache, nausea, dizziness, drowsiness, and un-coordination. Severe vision effects, including increased sensitivity to light, blurred vision, and blindness may develop following an 8-24 hour symptom-free period. Coma and death may result.

IRRITANT: Causes eye irritation. Aspiration hazard. Swallowing or vomiting of the liquid may result in aspiration (breathing) into the lungs.

POSSIBLE REPRODUCTIVE HAZARD: May cause fetotoxic (toxic to the fetus during the latter stages of pregnancy, often through the placenta) and teratogenic effects (causing malformations of the fetus), based on animal information.

NFPA Ratings: (Health, Fire, Reactivity): 1, 3, 0

3. Composition

Component	% (w/w)	Exposure Limits (ACGIH)*	LD_{50}	LC_{50}
Methanol (CAS 67-56-1)	99-100	ACGIH* TLV-TWA: 200 ppm, skin; TLV-STEL: 250 ppm, skin PEL-TWA: 200 ppm, skin PEL-STEL: 250 ppm, skin IDLH: 6000 ppm, acute inhalation toxicity to animals TLV Basis, critical effects: neuropathy, vision, central nervous system(CNS)	5628 mg/kg (oral/rat) 15800 mg/kg (dermal/ rabbit)	64000 ppm (inhalation/rat)

* Exposure limits may vary from time to time and from one jurisdiction to another. Check with local regulatory agency for the exposure limits in your area. ACGIH, American Conference of Governmental Industrial Hygienists.

4. First Aid Measures

Note: *Emergency assistance may also be available from the local poison control centre.*

Eye Contact: Remove contact lenses if worn. In case of contact, immediately flush eyes with plenty of clean running water for at least 15 minutes, lifting the upper and lower eyelids occasionally. Obtain medical attention.

Skin Contact: In case of contact, remove contaminated clothing. In a shower, wash affected areas with soap and water for at least 15 minutes. Seek medical attention if irritation occurs or persists. Wash clothing before reuse. Prolonged contact with methanol may defat skin tissue, resulting in drying and cracking.

Inhalation: Remove to fresh air, restore or assist breathing if necessary. Obtain medical attention.

Ingestion: Swallowing methanol is potentially life threatening. Onset of symptoms may be delayed for 18 to 24 hours after digestion. If conscious and medical aid is not immediately available, do not induce vomiting. In actual or suspected cases of ingestion, transport to medical facility immediately.

NOTE TO PHYSICIAN: Acute exposure to methanol, either through ingestion or breathing high airborne concentrations can result in symptoms appearing between 40 minutes and 72 hours after exposure. Symptoms and signs are usually limited to the Central Nervous System (CNS), eyes and gastrointestinal tract. Because of the initial CNS's effects of headache, vertigo, lethargy and confusion, there may be an impression of ethanol intoxication. Blurred vision, decreased acuity and photophobia are common complaints. Treatment with ipecac or lavage is indicated in any patient presenting within two hours of ingestion. A profound metabolic acidosis occurs in severe poisoning and serum bicarbonate levels are a

more accurate measure of severity than serum methanol levels. Treatment protocols are available from most major hospitals and early collaboration with appropriate hospitals is recommended.

Ethanol significantly decreases the toxicity of methanol because it competes for the same metabolic enzymes, and has been used to treat methanol poisoning.

5. Fire Fighting Measures

Suitable Extinguishing Media: Extinguishing Media: Small fires: Dry chemical, CO_2, water spray
Large fires: Water spray(see note in Unsuitable Extinguishing Media), AFFF(R) (Aqueous Film Forming Foam (alcohol resistant)) type with either a 3% or 6% foam proportioning system.

Unsuitable Extinguishing Media: General purpose synthetic foams or protein foams may work, but much less effectively. Water may be effective for cooling, but may not be effective for extinguishing a fire because it may not cool methanol below its flash point.

Specific Hazards: Methanol vapours may burn with an invisible flame. During a fire, carbon monoxide, carbon dioxide and irritation and toxic gases such as formaldehyde may be generated. Vapours can accumulate in confined spaces resulting in a toxicity and flammability hazard. Closed containers may rupture violently and suddenly release large quantities of methanol when exposed to fire or excessive heat for a sufficient period of time. Vapours are slightly heavier than air and may travel long distances toward sources of ignition.

Hazardous Combustion Products: Toxic gases and vapours; oxides of carbon and formaldehyde.

Fire Fighting Instructions: Methanol burns with a clean clear flame that is almost invisible in daylight. Stay upwind! Isolate and restrict area access. Concentrations of greater that 25% methanol in water can be ignited. Use fine water spray or fog to control fire spread and cool adjacent structures or containers. Contain fire control water for later disposal. Fire fighters must wear full face, positive pressure, self-contained breathing apparatus or airline and appropriate protective fire fighting clothing as per NFPA. Note that methanol fires may require proximity suits. Take care not to walk through any spilled chemical.

Special Information: Vapours can flow along surfaces to distant ignition sources and flash back.

6. Accidental Release Measures

Overview: Flammable liquid! Can burn without a visible flame. Release can cause an immediate risk of fire and explosion. Eliminate all ignition sources, stop leak and use absorbent materials. If necessary, contain spill by diking. Fluorocarbon alcohol resistant foams may be applied to spill to diminish vapour and fire hazard. Maximize methanol recovery for recycling or re-use. Restrict access to area until completion of cleanup. Ensure cleanup is conducted by trained personnel only. Wear adequate personal protection and remove all sources of ignition. Notify all governmental agencies as required by law.

Personal Protection: Full face, positive pressure self-contained breathing apparatus or airline, and fire resistant protective clothing with chemical resistant splash suit must be worn. If product ignites, approach and fire fighting must be done with appropriate fire fighting clothing.

Environmental Precautions: Biodegrades easily in water. Methanol in fresh or salt water may have serious effects on aquatic life. A study on methanol's toxic efffects on sewage sludge bacteria reported little effect on digestion at 0.1% while 0.5% methanol retarded digestion. Methanol will be broken down to carbon dioxide and water.

Remedial Measures: Flammable liquid. Release can cause an immediate fire/explosion hazard. Eliminate all sources of ignition, stop leak and use absorbent materials. Collect liquid with explosion proof pumps. Do not walk through spill product as it may be on fire and not visible.

Small Spills: Soak up spill with non-combustible absorbent material. Recover methanol and dilute with water to reduce fire hazard. Prevent spilled methanol from entering sewers, confined spaces, drains, or waterways. Restict access to unprotected personnel. Put material in suitable, covered, labeled containers. Flush area with water.

Large Spills: If necessary, contain spill by diking. Fluorocarbon alcohol resistant foams may be applied to spill to diminish vapour and fire hazard. Maximize methanol recovery for recycling or reuse. Collect liquid with explosion proof pumps.

7. Handling and Storage

Precautions for Handling: No smoking or open flame in storage, use or handling areas. Use explosion proof electrical equipment. Ensure proper electrical grounding procedures are in place.

Storage: Store in totally enclosed equipment, designed to avoid ignition and human contact. Tanks must be grounded, vented, and should have vapour emission controls. Tanks must be diked as per NFPA or API Standards. A flammable mixture of methanol vapour and air is possible inside a storage tank or transportation tank, and handlers should take appropriate precautions to reduce the risk of ignition. Handlers must eliminate ignition sources or purge the tank with an inert gas such as nitrogen. All equipment must be grounded - bonded when transferring product in order to avoid static discharge from the equipment, and subsequent possible fire. Avoid storage with incompatible materials. Anhydrous methanol is non-corrosive to most metals at ambient temperatures except for lead, nickel, monel, cast iron and high silicon iron. Coatings of copper (or copper alloys), zinc (including galvanized steel), or aluminum are unsuitable for storage. These materials may be attacked slowly by the methanol. Storage tanks of welded construction are normally satisfactory. They should be designed and built in conformance with good engineering practice for the material being stored. While plastics can be used for short term storage, they are generally not recommended for long-term storage due to deterioration effects and the subsequent risk of contamination.

Corrosion rates for several construction materials:

<0.508 mm/year:	Cast iron, monel, lead, nickel
<0.051 mm/year:	High silicon iron
Some attack:	Polyethylene
Satisfactory:	Neoprene, phenolic resins, polyesters, natural rubber, butyl rubber
Resistant:	Polyvinyl chloride, unplasticized

8. Exposure Controls, Personal Protection

Occupational Controls: ACGIH* TLV-TWA: 200 ppm, skin (262 mg/m^3);
TLV-STEL: 250 ppm, skin (328 mg/m^3);
PEL-TWA: 200 ppm, skin
PEL-STEL: 250 ppm, skin
TLV Basis: critical effects: neuropathy, vision, central nervous system(CNS)
IDLH: 6000 ppm, acute inhalation toxicity to animals

Engineering Controls: In confined areas, local and general ventilation should be provided to maintain airborne concentrations below permissable exposure limits. Ventilation systems must be designed according to approved engineering standards.

Respiratory Protection: NIOSH/OSHA recommendations for methanol concentrations in air:

Up to 2000 ppm: supplied air respirator
Up to 5000 ppm: supplied air respirator operated in a continuous-flow mode.
Up to 6000 ppm: supplied air respirator with a tight-fitting facepiece operated in a continuous- flow mode; or Full-facepiece self-contained breathing apparatus or Full-facepiece supplied air respirator.

Cartridge type respirators are NOT recommended.

Emergency or Planned entry into unkown concentrations or IDLH (immediately dangerous to life or health) conditions:

Respirator selection must be done by a qualified person and be based upon a risk assessment of the work activities and exposure levels. Respirators must be fit tested and users must be clean shaven where the respirator seals to the face. Exposure must be kept at or below the applicable exposure limits and the maximum use concentration of the respirator must not be exceeded.

Positive pressure, full-facepiece self-contained breathing apparatus; or Positive pressure, full-facepiece supplied air respirator with an auxiliary positive pressure self-contained breathing apparatus.

Skin Protection: Butyl and nitrile rubbers are recommended for gloves. Check with manufacturer. Wear chemical resistant pants and jackets, preferably of butyl or nitrile rubber. Check with manufacturer.

Eye and Face Protection: Face shield and chemical splash goggles when transferring is taking place. Contact lenses should not be worn when working with methanol.

Footwear: Chemical resistant and as specified by the workplace.

Other: Eyewash and showers should be located near work areas. NOTE: PPE must not be considered a long-term solution to exposure control. PPE usage must be accompanied by employer programs to properly select, maintain, clean, fit and use. Consult a competent industrial hygiene resource to determine hazard potential and/or the PPE manufacturers to ensure adequate protection.

Careful consideration must be made of the added danger of the concenentration being in the LEL/UEL range and so there may be a fire/explosion hazard.

9. Physical and Chemical Properties

Appearance: Liquid, clear, colourless
Odour: Mild characteristic alcohol odour
Odour Threshold: detection: 4.2 - 5960 ppm
(geometric mean) 160 ppm
recognition: 53 – 8940 ppm
(geometric mean) 690 ppm
pH: Not applicable
Freezing Point: -97.8°C
Boiling Point: 64.7°C
Boiling Range: Not determined
Flash Point: 11.0°C
Solubility: Completely soluble
Partial Coefficient: Log P (oct) = -0.82
Vapour Pressure: 12.8 kPa @ 20°C
Upper Explosive Limit (UEL): 36.5 %
Lower Explosive Limit (LEL): 6%
Auto Ignition Temperature: 464°C
Solvent Solubility: Soluble in all proportions in ethanol, benzene, other alcohols, chloroform, diethyl ether, other ethers, esters, ketones and most organic solvents
Critical Temperature: 239.4°C
Specific Gravity: 0.791 @ 20°C
Evaporation Rate: 4.1 (n-butyl acetate =1)
Vapour Density: 1.105 @ 15°C (air = 1)
Decomposition Temperature: Not determined
Sensitivity to Impact: No
Sensitivity to Static Charge: Low

10. Stability and Reactivity

Chemical Stability: Stable as supplied.

Hazardous Reactions: Yes. Avoid contact with strong oxidizers, strong mineral or organic acids, and strong bases. Contact with these materials may cause a violent or explosive reaction. May be corrosive to lead, aluminum, magnesium, and platinum.

Conditions to Avoid: Avoid contact with sparks, heat, open flame, or ignition sources.

Incompatibility: Yes. Avoid contact with strong oxidizers, strong mineral or organic acids, and strong bases. Contact with these materials may cause a violent or explosive reaction. May be corrosive to lead, aluminum, magnesium, and platinum. May react with metallic aluminum or magnesium and generate hydrogen gas. May attack some forms of plastic, rubber, and coatings.

Hazardous Decomposition Products: Formaldehyde, carbon dioxide, and carbon monoxide.

Hazardous Polymerization: Will not occur.

11. Toxicological Information

Signal Word/Label: DANGER! Extremely flammable liquid and vapour. Fatal if swallowed.
May damage fertility or the unborn child (fetotoxic and teratogenic effects).
May cause damage to eyes and central nervous system if ingested or inhaled.

Primary Routes of Entry:

Skin Contact:	Yes
Skin Absorption:	Yes
Eye Contact:	Yes
Ingestion:	Yes
Inhalation:	Yes

Emergency Overview: Colourless liquid, with a mild, characteristic alcohol odour when pure. Crude methanol may have a repulsive, pungent odour. Hygroscopic. Can decompose at high temperatures forming carbon monoxide and formaldehyde. Confined space toxicity hazard. Mild central nervous system depressant following inhalation, skin absorption or ingestion. May cause headache, nausea, dizziness, drowsiness, and incoordination. Severe vision effects, including increased sensitivity to light, blurred vision, and blindness may develop following an 8-24 hour symptom-free period. Coma and death may result. Causes eye irritation. Aspiration hazard. Swallowing or vomiting of the liquid may result in aspiration (breathing) into the lungs. May cause fetotoxic (toxic to the fetus during the latter stages of pregnancy, often through the placenta) and teratogenic effects (causing malformations of the fetus), based on animal information.

Acute Exposure:

Inhalation: Inhalation of high airborne concentrations can also irriate mucous membranes, cause headaches, sleepiness, nausea, confusion, loss of consciousness, digestive and visual disturbances and even death. NOTE: Odour threshhold of methanol is several times higher than the TLV-TWA. Depending upon severity of poisoning and the promptness of treatment, survivors may recover completely or may have permanent blindness, vision disturbances and/or nervous system effects. Concentrations in air exceeding 1000 ppm may cause irritation of the mucous membranes.

Skin Contact: Methanol is moderately irritating to the skin. Methanol can be absorbed through the skin and harmful effects have been reported by this route of entry. Effects are similar to those described in “Inhalation”.

Eye Contact: Methanol is a mild to moderate eye irritant. High vapour concentration or liquid contact with eyes causes irritation, tearing and burning.

Ingestion: Swallowing even small amounts of methanol could potentially cause blindness or death. Effects of sub lethal doses may be nausea, headache, abdominal pain, vomiting and visual disturbances ranging from blurred vision to light sensitivity.

Chronic Exposure:

Irritancy: Prolonged contact with skin may defat tissue causing dermititis or aggravate existing skin problems.

Sensitization: None reported.

Carcinogenicity: Not listed by IARC, NTP, ACGIH, or OSHA as a carcinogen.

Teratogenicity: Methanol has produced fetotoxicity in rats and teratogenicity in mice exposed by inhalation to high concentrations of methanol vapours **.**

Reproductive Toxicity: Information available does not suggest that methanol is a reproductive toxin.

Mutagenicity: There is insufficient information available to conclude that methanol is mutagenic.

Synergistic Products: In animals, high concentrations of methanol can increase the toxicity of other chemicals, particularly liver toxins like carbon tetrachloride. Ethanol significantly reduces the toxicity of methanol because it competes for the same metabolic enzymes, and has been usd to treat methanol poisoning.

Potential for Accumulation: Methanol is readily absorbed into the body following inhalation and ingestion. Skin absorption may occur if the skin is broken or exposure is prolonged. Once absorbed, methanol is rapidly distributed to body tissues. A small amount is excreted unchanged in exhaled air and the urine. The rest is first metabolized to formaldehyde, which is then metabolized to formic acid and/or formate. The formic acid and formate are eventually converted to carbon dioxide and water. In humans, methanol clears from the body, after inhalation or oral exposure, with a half-life of 1 day or more for high doses (greater than 1000 mg/kg) or about 1.5-3 hours for low doses (less than 100 mg/kg or 76.5-230 ppm (100-300 mg/m^3)).

Medical Conditions Aggravated By Exposure: Persons with pre-existing skin disorders, eye problems, respiratory conditions, or impaired liver or kidney functions may be more susceptable to the effects of this substance.

12. Ecological Information

Environmental toxicity: DO NOT discharge into sewer or waterways.

Methanol:

LC_{50} Pimephales promelas (fathead minnows) 29.4 g/L/96 hr, (28-29 days old), confidence limit= 28.5-30.4; Test conditions: Water temp= 25°C, dissolved oxygen= 7.3 mg/L, water hardness= 43.5 mg/l $CaCO_3$, alkalinity= 46.6 $CaCO_3$, tank volume= 6.3 L, additions= 5.71 V/D, pH= 7.66
LC_{50} Pimephales promelas (Fathead minnow, 28-32 day old, 0.126 g) 29,700 mg/L/24 hr; flow-through, 23.3+/-1.7°C, hardness 46.4 mg/L $CaCO_3$, pH 7.0-8.0
LC_{50} Pimephales promelas (Fathead minnow, 30 day old 0.12 g) 28,100 mg/L/96 hr; flow-through, 24-26°C, hardness 45.5 mg/L $CaCO_3$, pH 7.5
LC_{50} Daphnia pulex (Water flea, <24 hr old) 19,500 mg/L/18 hr; static, 22°C, hardness 23+/-2 mg/L $CaCO_3$
EC_{50} Daphnia obtusa (Water flea, <24 hr old; immobilization) 23,500 mg/L/24 hr; static, 20+/-2°C, hardness 250 mg/L $CaCO_3$, pH 7.8+/-0.2
EC_{50} Daphnia obtusa (Water flea, <24 hr old; immobilization) 22,200 mg/L/48 hr; static, 20+/-2°C, hardness 250 mg/L $CaCO_3$, pH 7.8+/-0.2

log K_{ow}: -0.82 – -0.66
Half-life (hr) air: 427
Half-life (hr) H_2O surface water: 5.3 – 64
Henry's Law constant (atm m^3/mol): $4.55X10^{-6}$
BOD 5 if unstated: 0.76 – 1.12
COD: 1.05 – 1.50, 99%
ThOD: 1.05
BCF: 0.2 – 10
TLm(48 hr): 8000mg/L (trout)
Toxicity Arthropoda: NOEL 10 g/L/48 hr (Daphnia)
HSNO Classification: 9.3C – Harmful to terrestrial vertebrates

Methanol in fresh or salt water may have serious effects on aquatic life. A study on methanol's toxic effects on sewage sludge bacteria reported little effect on digestion at 0.1% while 0.5% methanol retarded digestion. Methanol will be broken down into carbon dioxide and water.

Environmental Fate:

Biodegradability: Biodegrades easily in water and soil.

Bioaccumulation:

- **TERRESTRIAL FATE:** Based on a classification scheme, an estimated Koc value of 1 determined from a structure estimation method indicates that methanol is expected to have very high mobility in soil. Volatilization of methanol from moist soil surfaces is expected to be an important fate process given a Henry's Law constant of $4.55X10^{-6}$ (atm m^3/mol). The potential for volatilization of methanol from dry soil surfaces may exist based upon a vapor pressure of 127 mm Hg. Biodegradation is expected to be an important fate process for methanol.

- **AQUATIC FATE:** Based on a classification scheme, an estimated Koc value of 1, determined from a structure estimation method, indicates that methanol is not expected to adsorb to suspended solids and sediment. Volatilization from water surfaces is expected based upon a Henry's Law constant of $4.55X10^{-6}$ (atm m^3/mol). Using this Henry's Law constant and an estimation method, volatilization half-lives for a model river and model lake are three and 35 days, respectively. According to a classification scheme, a BCF of less than 10 measured in fish, suggests bioconcentration in aquatic organisms is low. Hydrolysis and photolysis in sunlit surface waters is not expected to be an important environmental fate process for methanol since this compound lacks functional groups that hydrolyze or absorb light under environmentally relevant conditions. Methanol has been shown to undergo rapid biodegradation in a variety of screening studies using sewage seed and activated sludge inoculum, which suggests that biodegradation will occur in aquatic environments.
- **ATMOSPHERIC FATE:** According to a model of gas/particle partitioning of semi volatile organic compounds in the atmosphere, methanol, which has a vapor pressure of 127 mm Hg at 25°C, is expected to exist solely as a vapor in the ambient atmosphere. Vapor-phase methanol is degraded in the atmosphere by reaction with photo chemically-produced hydroxyl radicals; the half-life for this reaction in air is estimated to be 17 days, calculated from its rate constant of $9.4X10^{-13}$ cu cm/molecule-sec at 25°C

13. Disposal Considerations

Review federal, provincial or state, and local government requirements prior to disposal. Store material for disposal as indicated in Section #7, ***Handling and Storage***. Disposal by controlled incineration or by secure land fill may be acceptable.

Recycle wherever possible. Large volumes may be suitable for re-distillation or, if contaminated, incinerated. Can be disposed of in a sewage treatment facility. Methanol levels of up to 0.1% act as a food source for bacteria; above this level may be toxic to bacteria. When pumping through sewage collection systems, the level of methanol should be kept below the flammable range (a 25% methanol/water mixture is non-flammable at temperatures below 39**°C**). 1 ppm of methanol is equivalent to 1.5 ppm BOD loading in the sewage plant.

Container disposal:

Empty containers may contain hazardous residue. Return to supplier for reuse if possible. Never weld, cut or grind empty containers. If disposing of containers, ensure they are well rinsed with water, then disposed of at an authorised landfill. After cleaning, all existing labels should be removed.

14. Transport Information

Canada Transportation of Dangerous Goods (TDG):	UN 1230, Methanol, Class 3(6.1), P.G. II Limited Quantity: ≤ 1 litres ERG Guide Number: 131
United States Department of Transport (49CFR): (Domestic Only)	UN 1230, Methanol, Class 3, P.G. II, (RQ 5000 lbs/2270 kg) Limited Quantity: ≤ 1 litres ERG Guide Number: 131
International Air Transport Association (IATA):	UN 1230, Methanol, Class 3(6.1), P.G. II Packaging Instruction (passenger aircraft): 305, 1 litre maximum per package,
International Maritime Organization (IMO):	UN 1230, Methanol, Class 3(6.1), P.G.II, Flash Point = 11°C EmS No. F-E, S-D Stowage Category "B", Clear of living quarters
Marine Pollutant:	No

15. Regulatory Information

CANADIAN FEDERAL REGULATIONS:

CEPA, DOMESTIC SUBSTANCES LIST: Listed (Canadian Environmental Protection Act (CEPA) Schedule I)

WHMIS CLASSIFICATION: B2, D1B, D2A, D2B

UNITED STATES REGULATIONS:

29CFR 1910.1200 (OSHA): Hazardous

40CFR 116-117 (EPA): Hazardous

40CFR 355, Appendices A and B: Subject to Emergency Planning and Notification

40CFR 372 (SARA Title III): Listed

40CFR 302 (CERCLA): Listed

TOXIC SUBSTANCES CONTROL ACT (TSCA): Listed in the inventory.

16. Other Information

References:

1. International Programme on Chemical Safety, Methanol, Environmental Health Criteria, World Health Organization 1997.
2. Patty's Industrial Hygiene and Toxicology, 5th Edition.
3. Fire Protection Guide to Hazardous Materials, 13th Edition.
4. Lanigan, S., Final report on the Safety Assessment of Methyl Alcohol, International Journal of Toxicology., Volume 20, Supplement 1 (2001).
5. Forsberg, K., Quick Selection Guide to Chemical Protective Clothing.
6. Nelson, B.K., Teratological assessment of Methanol and Ethanol at high inhalation levels in rats, Fundamental and Applied Toxicology, Volume 5.
7. NIOSH Guide to Chemical Hazards
8. Hazardous Substance Data Base (HSDB).
9. Cheminfo.

Original Preparation Date: September 22, 2005

Prepared by: Kel-Ex Agencies Ltd., P.O. Box 52201, Lynnmour RPO, North Vancouver, B.C., Canada, V7J 3V5

Disclaimer: The information above is believed to be accurate and represents the best information currently available to us. Users should make their own investigations to determine the suitability of the information for their particular purposes. This document is intended as a guide to the appropriate precautionary handling of the material by a properly trained person using this product.

Methanex Corporation and its subsidiaries make no representations or warranties, either express or implied, including without limitation any warranties of merchantability, fitness for a particular purpose with respect to the information set forth herein or the product to which the information refers. Accordingly, Methanex Corp. will not be responsible for damages resulting from use of or reliance upon this information.

This Material Safety Data Sheet may not be changed, or altered in any way without the expressed knowledge and permission of Methanex Corporation

Revisions: Revised and re-issued in GHS Format September 22, 2008

GFS CHEMICALS, INC.
P.O. Box 245 Powell, OH 43065
740-881-5501(Tel.) 740-881-5989(Fax)
1-800-424-9300(Chemtrec 24Hr. Info.)

MATERIAL SAFETY DATA SHEET

AmcoClear

AMCO CLEAR® TURBIDITY STANDARD

CHEMICAL NAME & SYNONYMS	**DOT CLASS**	**SARA TITLE 313**
AMCO CLEAR® Turbidity Standard	NR	No
	TSCA listed - Yes	

FORMULA	**REPORTABLE QUANTITY**	**F.W.**	**CAS#**
Styrene Divinyl Benzene Copolymer Beads <1%	N/A	N/A	9003-70-7
H_2O >99%	N/A	18.02	7732-18-5

PHYSICAL DATA
Boiling point 100°C; Density 1.0; melting point 0°C; pH 6.7

APPEARANCE & ODOR
White powder suspended in clear, colorless liquid. Depending on concentration, solution may be clear, hazy or opaque. Odorless.

REACTIVITY & CONDITIONS TO AVOID
Stable. Incompatible with organic matter (no hazardous reaction). Hazardous polymerization will not occur. Keep from freezing (once frozen, polymer will not remain completely suspended).

FIRE HAZARDS
None. NFPA # 0-0-0.

EXTINGUISHER	**FLASHPOINT**	**LEL**	**UEL**
Fight surrounding fire.	N/A	N/A	N/A

HEALTH HAZARDS
No health hazards by normal means of exposure. LD_{50} (oral-rabbit) 368 g (water)/kg. OSHA PEL/ACGIH TLV not established. No evidence of carcinogenicity.

SPECIAL PRECAUTIONS
Always use good laboratory practices. Keep from freezing, avoid contaminating solution.

FIRST AID
Flush eyes with water. Seek medical attention if irritation develops. Wash contacted skin with water. Ingestion is not hazardous. Inhalation is not an expected route of exposure.

SPILLS & LEAKS
Wash up with water. Flush to drain with plenty of water or general trash.

CATALOG #	**PREPARED BY**	**DATE**
Amco Clear	MDM	April 22, 2009

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P.O. Box 245 Powell, OH 43065
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MATERIAL SAFETY DATA SHEET

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EXTINGUISHER	**FLASHPOINT**	**LEL**	**UEL**
Fight surrounding fire.	N/A	N/A	N/A

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MATERIAL SAFETY DATA SHEET

AmcoClear

AMCO CLEAR® TURBIDITY STANDARD

CHEMICAL NAME & SYNONYMS	**DOT CLASS**	**SARA TITLE 313**
AMCO CLEAR® Turbidity Standard	NR	No
	TSCA listed - Yes	

FORMULA	**REPORTABLE QUANTITY**	**F.W.**	**CAS#**
Styrene Divinyl Benzene Copolymer Beads <1%	N/A	N/A	9003-70-7
H_2O >99%	N/A	18.02	7732-18-5

PHYSICAL DATA
Boiling point 100°C; Density 1.0; melting point 0°C; pH 6.7

APPEARANCE & ODOR
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REACTIVITY & CONDITIONS TO AVOID
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EXTINGUISHER	**FLASHPOINT**	**LEL**	**UEL**
Fight surrounding fire.	N/A	N/A	N/A

HEALTH HAZARDS
No health hazards by normal means of exposure. LD_{50} (oral-rabbit) 368 g (water)/kg. OSHA PEL/ACGIH TLV not established. No evidence of carcinogenicity.

SPECIAL PRECAUTIONS
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FIRST AID
Flush eyes with water. Seek medical attention if irritation develops. Wash contacted skin with water. Ingestion is not hazardous. Inhalation is not an expected route of exposure.

SPILLS & LEAKS
Wash up with water. Flush to drain with plenty of water or general trash.

CATALOG #	**PREPARED BY**	**DATE**
Amco Clear	MDM	April 22, 2009

OAKTON® MATERIAL SAFETY DATA SHEET

Section 1. Chemical Product and Company Identification

Catalog Number(s)

00654-08, 35654-08, 05942-61, 05942-62, 05942-64, 05942-65, 05942-66, 05942-67, 35653-03

Product Identity

BUFFER, Standard, pH 10.00; BUFFER, High Accuracy, pH 10.000 (Color Coded Blue)

Manufacturer's Name RICCA CHEMICAL COMPANY	Emergency Telephone Number (24 hr) CHEMTREC®: 800-424-9300
Address (Number, Street, City, State, and ZIP Code) P.O. Box 13090	Telephone Number For Information 817-461-5601
Arlington, Texas 76094	Date Prepared 3-8-2000

Section 2. Composition / Information on Ingredients

Component	CAS Registry #	Percent Concentration	Exposure Limits ACGIH TLV	OSHA PEL
Sodium Carbonate	497-19-8	< 1	N/A	N/A
Sodium Bicarbonate	144-55-8	< 1	N/A	N/A
Preservative* *(No Mercury compounds or Formaldehyde)	proprietary	< 0.1	N/A	N/A
Inert Dye	proprietary	< 0.1	N/A	N/A
Water, Deionized	7732-18-5	Balance	N/A	N/A

Section 3. Hazards Identification

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EMERGENCY OVERVIEW

Non-flammable, non-toxic, non-corrosive. Does not present any significant health hazards. Wash areas of contact with water.

☆☆☆

POTENTIAL HEALTH EFFECTS:

TARGET ORGANS: eyes, skin.

EYE CONTACT: May cause slight irritation.

INHALATION: Not likely to be hazardous by inhalation.

SKIN CONTACT: May cause slight irritation.

INGESTION: Large doses may cause nausea, vomiting, diarrhea and cramps.

CHRONIC EFFECTS / CARCINOGENICITY:

IARC – No
NTP – No
OSHA – No

TERATOLOGY (BIRTH DEFECT) INFORMATION:

Mutation data cited in "Registry of Toxic Effects of Chemical Substances" for Sodium Bicarbonate in rats.

REPRODUCTION INFORMATION:

Reproductive data cited in "Registry of Toxic Effects of Chemical Substances" for Sodium Bicarbonate and Sodium Carbonate in mice.

Section 4. First Aid Measures – In all cases, seek qualified evaluation.

EYE CONTACT: Irrigate immediately with large quantity of water for at least 15 minutes. Call a physician if irritation develops.

INHALATION: Remove to fresh air. Give artificial respiration if necessary. If breathing is difficult, give oxygen.

SKIN CONTACT: Flush with plenty of water for at least 15 minutes. Call a physician if irritation develops.

INGESTION: Dilute with water or milk. Call a physician if necessary.

Section 5. Fire Fighting Measures

FLAMMABLE PROPERTIES:

FLASH POINT: N/A METHOD USED: N/A

FLAMMABLE LIMITS

LFL: N/A UFL: N/A

EXTINGUISHING MEDIA: Use any means suitable for extinguishing surrounding fire.

FIRE & EXPLOSION HAZARDS: Not considered to be a fire or explosion hazard.

FIRE FIGHTING INSTRUCTIONS: Use normal procedures/instructions.

FIRE FIGHTING EQUIPMENT: Use protective clothing and breathing equipment appropriate for the surrounding fire.

Section 6. Accidental Release Measures

Absorb with suitable material and treat as normal refuse. Small amounts of the liquid may be flushed to the drain with excess water. Always dispose of in accordance with local regulations.

Section 7. Handling and Storage

As with all chemicals, wash hands thoroughly after handling. Avoid contact with eyes and skin. Protect from freezing and physical damage. SAFETY STORAGE CODE: GENERAL

Section 8. Exposure Controls / Personal Protection

ENGINEERING CONTROLS: No specific controls are needed. Normal room ventilation is adequate.

RESPIRATORY PROTECTION: Normal room ventilation is adequate.

SKIN PROTECTION: Chemical resistant gloves.

EYE PROTECTION: Safety glasses or goggles.

Section 9. Physical and chemical Properties

APPEARANCE:	Clear, blue colored liquid	**pH:**	10
ODOR:	Odorless	**BOILING POINT (^{o}C):**	approximately 100
SOLUBILITY IN WATER:	Infinite	**MELTING POINT (^{o}C):**	approximately 0
SPECIFIC GRAVITY:	approximately 1	**VAPOR PRESSURE:**	N/A

Section 10. Stability and Reactivity

CHEMICAL STABILITY: Stable under normal conditions of use and storage.

INCOMPATIBILITY: Acids

HAZARDOUS DECOMPOSITION PRODUCTS: Oxides of Sodium.

HAZARDOUS POLYMERIZATION: Will not occur.

Section 11. Toxicological Information

LD50, Oral, Rat: 4090 mg/kg (Sodium Carbonate), 4220 mg/kg (Sodium Bicarbonate), details of toxic effects not reported other than lethal dose value.

Section 12. Ecological Information

ECOTOXICOLOGICAL INFORMATION: No information found.

CHEMICAL FATE INFORMATION: No information found.

Section 13. Disposal Considerations

Dilute with water, then flush to sewer if local regulations allow. If not allowed, save for recovery or recycling in an approved waste disposal facility. Always dispose of in accordance with local, state and federal regulations.

Section 14. Transport Information (Not meant to be all inclusive)

D.O.T. SHIPPING NAME:	Not regulated
D.O.T. HAZARD CLASS:	None
U.N. / N.A. NUMBER:	None
PACKING GROUP:	None
D.O.T. LABEL:	None

Section 15. Regulatory Information (Not meant to be all inclusive - selected regulation represented)

OSHA STATUS: The above items either do not contain any specifically hazardous material or the potentially hazardous material is present in such low concentration that the items do not present any immediate threat to health and safety. These items do not meet the OSHA Hazard Communication Standard (29 CFR 1910.1200) definition of a hazardous material.

TSCA STATUS: All components of this solution are listed on the TSCA Inventory.

CERCLA REPORTABLE QUANTITY: Not reportable

SARA TITLE III:

SECTION 302 EXTREMELY HAZARDOUS SUBSTANCES: No

SECTION 311/312 HAZARDOUS CATEGORIES: No

SECTION 313 TOXIC CHEMICALS: No

RCRA STATUS: No

CALIFORNIA PROPOSITION 65: Not listed.

Section 16. Other Information

NFPA® Ratings:	**Health: 1**	**Flammability: 0**	**Reactivity: 0**	**Special Notice Key: None**
HMIS® Ratings:	**Health: 1**	**Flammability: 0**	**Reactivity: 0**	**Protective Equipment: B (Protective eyewear, gloves)**

Rev 1, 01-15-2003: added catalog number 35653-03.
Rev 2, 03-25-2003: Reviewed and approved.
Rev 3, 03-20-2006: Reviewed and approved.

When handled properly by qualified personnel, the product described herein does not present a significant health or safety hazard. Alteration of its characteristics by concentration, evaporation, addition of other substances, or other means may present hazards not specifically addressed herein and

which must be evaluated by the user. The information furnished herein is believed to be accurate and represents the best data currently available to us. No warranty, expressed or implied, is made and RICCA CHEMICAL COMPANY assumes no legal responsibility or liability whatsoever resulting from its use.

OAKTON® MATERIAL SAFETY DATA SHEET

Section 1. Chemical Product and Company Identification

Catalog Number(s)

00654-00, 05942-21, 05942-22, 05942-24, 05942-25, 05942-26, 05942-27, 35653-01, 35654-00

Product Identity

BUFFER, Standard, pH 4.01; BUFFER, High Accuracy, pH 4.000 (Color Coded Red)

Manufacturer's Name	Emergency Telephone Number (24 hr)
RICCA CHEMICAL COMPANY	CHEMTREC®: 800-424-9300
Address (Number, Street, City, State, and ZIP Code)	Telephone Number For Information
P.O. Box 13090	817-461-5601
	Date Prepared
Arlington, Texas 76094	3-7-2000

Section 2. Composition / Information on Ingredients

Component	CAS Registry #	Percent Concentration	Exposure Limits ACGIH TLV	Exposure Limits OSHA PEL
Potassium Acid Phthalate	877-24-7	0.95 – 1.05	N/A	N/A
Preservative* *(No Mercury compounds or Formaldehyde)	proprietary	<0.5	N/A	N/A
Inert Dye	proprietary	<0.1	N/A	N/A
Water, Deionized	7732-18-5	Balance	N/A	N/A

Section 3. Hazards Identification

☆☆☆

Section 4. First Aid Measures – In all cases, seek qualified evaluation.

EYE CONTACT: Irrigate immediately with large quantity of water for at least 15 minutes. Call a physician if irritation develops.

INHALATION: Remove to fresh air. Give artificial respiration if necessary. If breathing is difficult, give oxygen.

SKIN CONTACT: Flush with plenty of water for at least 15 minutes. Call a physician if irritation develops.

INGESTION: Dilute with water or milk. Call a physician if necessary.

Section 5. Fire Fighting Measures

FLAMMABLE PROPERTIES:

FLASH POINT: N/A METHOD USED: N/A

FLAMMABLE LIMITS

LFL: N/A UFL: N/A

EXTINGUISHING MEDIA: Use any means suitable for extinguishing surrounding fire.

FIRE & EXPLOSION HAZARDS: Not considered to be a fire or explosion hazard.

FIRE FIGHTING INSTRUCTIONS: Use normal procedures/instructions.

FIRE FIGHTING EQUIPMENT: Use protective clothing and breathing equipment appropriate for the surrounding fire.

Section 6. Accidental Release Measures

Absorb with suitable material and dispose of in accordance with local regulations.

Section 7. Handling and Storage

As with all chemicals, wash hands thoroughly after handling. Avoid contact with eyes and skin. Protect from freezing and physical damage. SAFETY STORAGE CODE: GENERAL

Section 8. Exposure Controls / Personal Protection

ENGINEERING CONTROLS: No specific controls are needed. Normal room ventilation is adequate.

RESPIRATORY PROTECTION: Normal room ventilation is adequate.

SKIN PROTECTION: Chemical resistant gloves.

EYE PROTECTION: Safety glasses or goggles.

Section 9. Physical and chemical Properties

APPEARANCE:	Clear, red colored liquid	**pH:**	4
ODOR:	odorless	**BOILING POINT (^{o}C):**	approximately 100
SOLUBILITY IN WATER:	infinite	**MELTING POINT (^{o}C):**	approximately 0
SPECIFIC GRAVITY:	approximately 1	**VAPOR PRESSURE:**	N/A

Section 10. Stability and Reactivity

CHEMICAL STABILITY: Stable under normal conditions of use and storage.

INCOMPATIBILITY: Nitric Acid

HAZARDOUS DECOMPOSITION PRODUCTS: Oxides of Carbon and Potassium.

HAZARDOUS POLYMERIZATION: Will not occur.

Section 11. Toxicological Information

LD50, Oral, Rat: >3200 mg/kg (Potassium Acid Phthalate), details of toxic effects not reported other than lethal dose value.

Section 12. Ecological Information

ECOTOXICOLOGICAL INFORMATION: No information found.

CHEMICAL FATE INFORMATION: No information found.

Section 13. Disposal Considerations

Dilute with water, neutralize with weak sodium hydroxide solution, and then flush to sewer if local regulations allow. If not allowed, save for recovery or recycling in an approved waste disposal facility. Always dispose of in accordance with local, state and federal regulations.

Section 14. Transport Information (Not meant to be all inclusive)

D.O.T. SHIPPING NAME:	Not regulated
D.O.T. HAZARD CLASS:	None
U.N. / N.A. NUMBER:	None
PACKING GROUP:	None
D.O.T. LABEL:	None

Section 15. Regulatory Information (Not meant to be all inclusive - selected regulation represented)

OSHA STATUS: The above items either do not contain any specifically hazardous material or the potentially hazardous material is present in such low concentration that the items do not present any immediate threat to health and safety. These items do not meet the OSHA Hazard Communication Standard (29 CFR 1910.1200) definition of a hazardous material.

TSCA STATUS: All components of this solution are listed on the TSCA Inventory.
CERCLA REPORTABLE QUANTITY: Not reportable
SARA TITLE III:

SECTION 302 EXTREMELY HAZARDOUS SUBSTANCES: No
SECTION 311/312 HAZARDOUS CATEGORIES: No
SECTION 313 TOXIC CHEMICALS: No

RCRA STATUS: No

CALIFORNIA PROPOSITION 65: Not listed

Section 16. Other Information

NFPA® Ratings:	**Health: 1**	**Flammability: 0**	**Reactivity: 0**	**Special Notice Key: None**
HMIS® Ratings:	**Health: 1**	**Flammability: 0**	**Reactivity: 0**	**Protective Equipment: B (Protective eyewear, gloves)**

Rev 1, 10-16-2000: (Section 1) added catalog number 35653-01.
Rev 2, 03-25-2003: Reviewed and approved.
Rev 3, 03-20-2006: Reviewed and approved.

When handled properly by qualified personnel, the product described herein does not present a significant health or safety hazard. Alteration of its characteristics by concentration, evaporation, addition of other substances, or other means may present hazards not specifically addressed herein and which must be evaluated by the user. The information furnished herein is believed to be accurate and represents the best data currently available to us. No warranty, expressed or implied, is made and RICCA CHEMICAL COMPANY assumes no legal responsibility or liability whatsoever resulting from its use.

OAKTON® MATERIAL SAFETY DATA SHEET

Section 1. Chemical Product and Company Identification

Catalog Number(s)

00654-04, 35654-04, 05942-41, 05942-42, 05942-44, 05942-45, 35653-02

Product Identity

BUFFER, Standard, pH 7.00 (Color Coded Green)

Manufacturer's Name	Emergency Telephone Number (24 hr)
RICCA CHEMICAL COMPANY	CHEMTREC®: 800-424-9300
Address (Number, Street, City, State, and ZIP Code)	Telephone Number For Information
P.O. Box 13090	817-461-5601
	Date Prepared
Arlington, Texas 76094	3-8-2000

Section 2. Composition / Information on Ingredients

Component	CAS Registry #	Percent Concentration	Exposure Limits ACGIH TLV	Exposure Limits OSHA PEL
Sodium Phosphate, Dibasic	7558-79-4	< 1	N/A	N/A
Potassium Phosphate, Monobasic	7778-77-0	< 1	N/A	N/A
Preservative* *(No Mercury Compounds or Formaldehyde)	Proprietary	< 0.1	N/A	N/A
Inert Dye	Proprietary	< 0.1	N/A	N/A
Water, Deionized	7732-18-5	Balance	N/A	N/A

Section 3. Hazards Identification

☆☆☆

Section 4. First Aid Measures – In all cases, seek qualified evaluation.

EYE CONTACT: Irrigate immediately with large quantity of water for at least 15 minutes. Call a physician if irritation develops.

INHALATION: Remove to fresh air. Give artificial respiration if necessary. If breathing is difficult, give oxygen.

SKIN CONTACT: Flush with plenty of water for at least 15 minutes. Call a physician if irritation develops.

INGESTION: Dilute with water or milk. Call a physician if necessary.

Section 5. Fire Fighting Measures

FLAMMABLE PROPERTIES:

FLASH POINT: N/A METHOD USED: N/A

FLAMMABLE LIMITS

LFL: N/A UFL: N/A

EXTINGUISHING MEDIA: Use any means suitable for extinguishing surrounding fire.

FIRE & EXPLOSION HAZARDS: Not considered to be a fire or explosion hazard.

FIRE FIGHTING INSTRUCTIONS: Use normal procedures/instructions.

FIRE FIGHTING EQUIPMENT: Use protective clothing and breathing equipment appropriate for the surrounding fire.

Section 6. Accidental Release Measures

Absorb with suitable material (vermiculite, clay, etc.) and dispose of in accordance with local regulations. Check with local agencies for the proper disposal of phosphate containing solutions.

Section 7. Handling and Storage

As with all chemicals, wash hands thoroughly after handling. Avoid contact with eyes and skin. Protect from freezing and physical damage. SAFETY STORAGE CODE: GENERAL

Section 8. Exposure Controls / Personal Protection

ENGINEERING CONTROLS: No specific controls are needed. Normal room ventilation is adequate.

RESPIRATORY PROTECTION: Normal room ventilation is adequate.

SKIN PROTECTION: Chemical resistant gloves.

EYE PROTECTION: Safety glasses or goggles.

Section 9. Physical and chemical Properties

APPEARANCE:	Clear, green liquid	**pH:**	7
ODOR:	Odorless	**BOILING POINT (^{o}C):**	approximately 100
SOLUBILITY IN WATER:	Infinite	**MELTING POINT (^{o}C):**	approximately 0
SPECIFIC GRAVITY:	approximately 1	**VAPOR PRESSURE:**	N/A

Section 10. Stability and Reactivity

CHEMICAL STABILITY: Stable under normal conditions of use and storage.

INCOMPATIBILITY: None identified.

HAZARDOUS DECOMPOSITION PRODUCTS: Phosphorus oxides may form when heated to decomposition.

HAZARDOUS POLYMERIZATION: Will not occur.

Section 11. Toxicological Information

LD50, Oral, Rat: (Sodium Phosphate Dibasic) 17 gm/kg; LD50, Dermal, Rabbit: (Potassium Phosphate Monobasic) >4640 mg/kg; details of toxic effects not reported other than lethal dose value.

Section 12. Ecological Information

ECOTOXICOLOGICAL INFORMATION: No information found.

CHEMICAL FATE INFORMATION: No information found.

Section 13. Disposal Considerations

Dilute with water, then flush to sewer if local regulations allow for the flushing of phosphate containing solutions. If not allowed, save for recovery or recycling in an approved waste disposal facility. Always dispose of in accordance with local, state and federal regulations.

Section 14. Transport Information (Not meant to be all inclusive)

D.O.T. SHIPPING NAME:	Not regulated
D.O.T. HAZARD CLASS:	None
U.N. / N.A. NUMBER:	None
PACKING GROUP:	None
D.O.T. LABEL:	None

Section 15. Regulatory Information (Not meant to be all inclusive - selected regulation represented)

OSHA STATUS: The above items either do not contain any specifically hazardous material or the potentially hazardous material is present in such low concentration that the items do not present any immediate threat to health and safety. These items do not meet the OSHA Hazard Communication Standard (29 CFR 1910.1200) definition of a hazardous material.

TSCA STATUS: All components of this solution are listed on the TSCA Inventory or are mixtures (hydrates) of items listed on the TSCA Inventory.

CERCLA REPORTABLE QUANTITY: Sodium Phosphate, Dibasic - 5,000 pounds.

SARA TITLE III:

SECTION 302 EXTREMELY HAZARDOUS SUBSTANCES: No

SECTION 311/312 HAZARDOUS CATEGORIES: No

SECTION 313 TOXIC CHEMICALS: No

RCRA STATUS: No

CALIFORNIA PROPOSITION 65: Not listed.

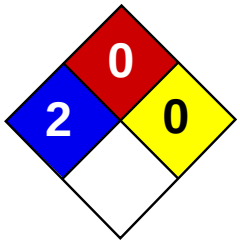
PENNSYLVANIA: Sodium Phosphate Dibasic is listed as an environmental hazard on the state Hazardous Substance list.

Section 16. Other Information

NFPA Ratings:	**Health: 1**	**Flammability: 0**	**Reactivity: 0**	**Special Notice Key: None**
HMIS® Ratings:	**Health: 1**	**Flammability: 0**	**Reactivity: 0**	**Protective Equipment: B (Protective eyewear, gloves)**

Rev 1, 8-25-2000: (Section 2) corrected concentration of preservative from 1 – 2 to < 0.1%.
Rev 2, 03-25-2003: Reviewed and approved, (Section 15) added CERCLA reportable quantity.
Rev 3, 03-20-2006: Reviewed and approved.

When handled properly by qualified personnel, the product described herein does not present a significant health or safety hazard. Alteration of its characteristics by concentration, evaporation, addition of other substances, or other means may present hazards not specifically addressed herein and which must be evaluated by the user. The information furnished herein is believed to be accurate and represents the best data currently available to us. No warranty, expressed or implied, is made and RICCA CHEMICAL COMPANY assumes no legal responsibility or liability whatsoever resulting from its use.



Material Safety Data Sheet
Tetrachloroethylene MSDS

Section 1: Chemical Product and Company Identification

Product Name: Tetrachloroethylene

Catalog Codes: SLT3220

CAS#: 127-18-4

RTECS: KX3850000

TSCA: TSCA 8(b) inventory: Tetrachloroethylene

CI#: Not available.

Synonym: Perchloroethylene; 1,1,2,2-Tetrachloroethylene; Carbon bichloride; Carbon dichloride; Ankilostin; Didakene; Dilatin PT; Ethene, tetrachloro-; Ethylene tetrachloride; Perawin; Perchlor; Perclene; Perclene D; Percosolvel; Tetrachloroethene; Tetraleno; Tetralex; Tetravec; Tetroguer; Tetropil

Chemical Name: Ethylene, tetrachloro-

Chemical Formula: C2-Cl4

Contact Information:

Sciencelab.com, Inc.
14025 Smith Rd.
Houston, Texas 77396

US Sales: **1-800-901-7247**
International Sales: **1-281-441-4400**

Order Online: ScienceLab.com

CHEMTREC (24HR Emergency Telephone), call: 1-800-424-9300

International CHEMTREC, call: 1-703-527-3887

For non-emergency assistance, call: 1-281-441-4400

Section 2: Composition and Information on Ingredients

Composition:

Name	CAS #	% by Weight
Tetrachloroethylene	127-18-4	100

Toxicological Data on Ingredients: Tetrachloroethylene: ORAL (LD50): Acute: 2629 mg/kg [Rat]. DERMAL (LD): Acute: >3228 mg/kg [Rabbit]. MIST(LC50): Acute: 34200 mg/m 8 hours [Rat]. VAPOR (LC50): Acute: 5200 ppm 4 hours [Mouse].

Section 3: Hazards Identification

Potential Acute Health Effects:
Hazardous in case of skin contact (irritant), of inhalation. Slightly hazardous in case of skin contact (permeator), of eye contact (irritant), of ingestion.

Potential Chronic Health Effects:
CARCINOGENIC EFFECTS: Classified A3 (Proven for animal.) by ACGIH. Classified 2A (Probable for human.) by IARC, 2 (anticipated carcinogen) by NTP. MUTAGENIC EFFECTS: Mutagenic for bacteria and/or yeast. TERATOGENIC EFFECTS: Not available. DEVELOPMENTAL TOXICITY: Not available. The substance may be toxic to kidneys, liver, peripheral nervous system, respiratory tract, skin, central nervous system (CNS). Repeated or prolonged exposure to the substance can produce target organs damage.

Section 4: First Aid Measures

Eye Contact:
Check for and remove any contact lenses. In case of contact, immediately flush eyes with plenty of water for at least 15 minutes. Get medical attention if irritation occurs.

Skin Contact:
In case of contact, immediately flush skin with plenty of water. Cover the irritated skin with an emollient. Remove contaminated clothing and shoes. Wash clothing before reuse. Thoroughly clean shoes before reuse. Get medical attention.

Serious Skin Contact:
Wash with a disinfectant soap and cover the contaminated skin with an anti-bacterial cream. Seek medical attention.

Inhalation:
If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Get medical attention if symptoms appear.

Serious Inhalation:
Evacuate the victim to a safe area as soon as possible. Loosen tight clothing such as a collar, tie, belt or waistband. If breathing is difficult, administer oxygen. If the victim is not breathing, perform mouth-to-mouth resuscitation. Seek medical attention.

Ingestion:
Do NOT induce vomiting unless directed to do so by medical personnel. Never give anything by mouth to an unconscious person. Loosen tight clothing such as a collar, tie, belt or waistband. Get medical attention if symptoms appear.

Serious Ingestion: Not available.

Section 5: Fire and Explosion Data

Flammability of the Product: Non-flammable.

Auto-Ignition Temperature: Not applicable.

Flash Points: Not applicable.

Flammable Limits: Not applicable.

Products of Combustion: Not available.

Fire Hazards in Presence of Various Substances: Not applicable.

Explosion Hazards in Presence of Various Substances:
Risks of explosion of the product in presence of mechanical impact: Not available. Risks of explosion of the product in presence of static discharge: Not available.

Fire Fighting Media and Instructions: Not applicable.

Special Remarks on Fire Hazards: Not available.

Special Remarks on Explosion Hazards: Not available.

Section 6: Accidental Release Measures

Small Spill: Absorb with an inert material and put the spilled material in an appropriate waste disposal.

Large Spill:
Absorb with an inert material and put the spilled material in an appropriate waste disposal. Be careful that the product is not present at a concentration level above TLV. Check TLV on the MSDS and with local authorities.

Section 7: Handling and Storage

Precautions:
Do not ingest. Do not breathe gas/fumes/ vapor/spray. Avoid contact with skin. Wear suitable protective clothing. In case of insufficient ventilation, wear suitable respiratory equipment. If ingested, seek medical advice immediately and show the container or the label. Keep away from incompatibles such as oxidizing agents, metals, acids, alkalis.

Storage: Keep container tightly closed. Keep container in a cool, well-ventilated area.

Section 8: Exposure Controls/Personal Protection

Engineering Controls:
Provide exhaust ventilation or other engineering controls to keep the airborne concentrations of vapors below their respective threshold limit value.

Personal Protection:
Safety glasses. Lab coat. Vapor respirator. Be sure to use an approved/certified respirator or equivalent. Gloves.

Personal Protection in Case of a Large Spill:
Splash goggles. Full suit. Vapor respirator. Boots. Gloves. A self contained breathing apparatus should be used to avoid inhalation of the product. Suggested protective clothing might not be sufficient; consult a specialist BEFORE handling this product.

Exposure Limits:
TWA: 25 (ppm) from OSHA (PEL) [United States] TWA: 25 STEL: 100 (ppm) from ACGIH (TLV) [United States] TWA: 170 (mg/m3) from OSHA (PEL) [United States] Consult local authorities for acceptable exposure limits.

Section 9: Physical and Chemical Properties

Physical state and appearance: Liquid.

Odor: Ethereal.

Taste: Not available.

Molecular Weight: 165.83 g/mole

Color: Clear Colorless.

pH (1% soln/water): Not available.

Boiling Point: 121.3°C (250.3°F)

Melting Point: -22.3°C (-8.1°F)

Critical Temperature: 347.1°C (656.8°F)

Specific Gravity: 1.6227 (Water = 1)

Vapor Pressure: 1.7 kPa (@ 20°C)

Vapor Density: 5.7 (Air = 1)

Volatility: Not available.

Odor Threshold: 5 - 50 ppm

Water/Oil Dist. Coeff.: The product is more soluble in oil; log(oil/water) = 3.4

Ionicity (in Water): Not available.

Dispersion Properties: Not available.

Solubility:
Miscible with alcohol, ether, chloroform, benzene, hexane. It dissolves in most of the fixed and volatile oils. Solubility in water: 0.015 g/100 ml @ 25 deg. C It slowly decomposes in water to yield Trichloroacetic and Hydrochloric acids.

Section 10: Stability and Reactivity Data

Stability: The product is stable.

Instability Temperature: Not available.

Conditions of Instability: Incompatible materials

Incompatibility with various substances: Reactive with oxidizing agents, metals, acids, alkalis.

Corrosivity: Non-corrosive in presence of glass.

Special Remarks on Reactivity:
Oxidized by strong oxidizing agents. Incompatible with sodium hydroxide, finely divided or powdered metals such as zinc, aluminum, magnesium, potassium, chemically active metals such as lithium, beryllium , barium. Protect from light.

Special Remarks on Corrosivity: Slowly corrodes aluminum, iron, and zinc.

Polymerization: Will not occur.

Section 11: Toxicological Information

Routes of Entry: Absorbed through skin. Eye contact. Inhalation. Ingestion.

Toxicity to Animals:
WARNING: THE LC50 VALUES HEREUNDER ARE ESTIMATED ON THE BASIS OF A 4-HOUR EXPOSURE. Acute oral toxicity (LD50): 2629 mg/kg [Rat]. Acute dermal toxicity (LD50): >3228 mg/kg [Rabbit]. Acute toxicity of the vapor (LC50): 5200 4 hours [Mouse].

Chronic Effects on Humans:
CARCINOGENIC EFFECTS: Classified A3 (Proven for animal.) by ACGIH. Classified 2A (Probable for human.) by IARC, 2 (Some evidence.) by NTP. MUTAGENIC EFFECTS: Mutagenic for bacteria and/or yeast. May cause damage to the following organs: kidneys, liver, peripheral nervous system, upper respiratory tract, skin, central nervous system (CNS).

Other Toxic Effects on Humans:
Hazardous in case of skin contact (irritant), of inhalation. Slightly hazardous in case of skin contact (permeator), of ingestion.

Special Remarks on Toxicity to Animals:
Lowest Publishe Lethal Dose/Conc: LDL [Rabbit] - Route: Oral; Dose: 5000 mg/kg LDL [Dog] - Route: Oral; Dose: 4000 mg/kg LDL [Cat] - Route: Oral; Dose: 4000 mg/kg

Special Remarks on Chronic Effects on Humans:
May cause adverse reproductive effects and birth defects(teratogenic). May affect genetic material (mutagenic). May cause cancer.

Special Remarks on other Toxic Effects on Humans:
Acute Potential Health Effects: Skin: Causes skin irritation with possible dermal blistering or burns. Symtoms may include redness, itching, pain, and possible dermal blistering or burns. It may be absorbed through the skin with possible systemic effects. A single prolonged skin exposure is not likely to result in the material being absorbed in harmful amounts. Eyes: Contact causes transient eye irritation, lacrimation. Vapors cause eye/conjunctival irritation. Symptoms may include redness and pain. Inhalation: The main route to occupational exposure is by inhalation since it is readily absorbed through the lungs. It causes respiratory tract irritation, . It can affect behavior/central nervous system (CNS depressant and anesthesia ranging from slight inebriation to death, vertigo, somnolence, anxiety, headache, excitement, hallucinations, muscle incoordination, dizziness, lightheadness, disorentiation, seizures, enotional instability, stupor, coma). It may cause pulmonary edema Ingestion: It can cause nausea, vomiting, anorexia, diarrhea, bloody stool. It may affect the liver, urinary system (proteinuria, hematuria, renal failure, renal tubular disorder), heart (arrhythmias). It may affect behavior/central nervous system with symptoms similar to that of inhalation. Chronic Potential Health Effects: Skin: Prolonged or repeated skin contact may result in excessive drying of the skin, and irritation. Ingestion/Inhalation: Chronic exposure can affect the liver(hepatitis,fatty liver degeneration), kidneys, spleen, and heart (irregular heartbeat/arrhythmias, cardiomyopathy, abnormal EEG), brain, behavior/ central nervous system/peripheral nervous system (impaired memory, numbness of extremeties, peripheral neuropathy and other

Section 12: Ecological Information

Ecotoxicity:
Ecotoxicity in water (LC50): 18.4 mg/l 96 hours [Fish (Fatthead Minnow)]. 18 mg/l 48 hours [Daphnia (daphnia)]. 5 mg/l 96 hours [Fish (Rainbow Trout)]. 13 mg/l 96 hours [Fish (Bluegill sunfish)].

BOD5 and COD: Not available.

Products of Biodegradation:
Possibly hazardous short term degradation products are not likely. However, long term degradation products may arise.

Toxicity of the Products of Biodegradation: The product itself and its products of degradation are not toxic.

Special Remarks on the Products of Biodegradation: Not available.

Section 13: Disposal Considerations

Waste Disposal:
Waste must be disposed of in accordance with federal, state and local environmental control regulations.

Section 14: Transport Information

DOT Classification: CLASS 6.1: Poisonous material.

Identification: : Tetrachloroethylene UNNA: 1897 PG: III

Special Provisions for Transport: Marine Pollutant

Section 15: Other Regulatory Information

Federal and State Regulations:
California prop. 65: This product contains the following ingredients for which the State of California has found to cause cancer, birth defects or other reproductive harm, which would require a warning under the statute: Tetrachloroethylene California prop. 65: This product contains the following ingredients for which the State of California has found to cause cancer which would require a warning under the statute: Tetrachloroethylene Connecticut hazardous material survey.: Tetrachloroethylene Illinois toxic substances disclosure to employee act: Tetrachloroethylene Illinois chemical safety act: Tetrachloroethylene New York release reporting list: Tetrachloroethylene Rhode Island RTK hazardous substances: Tetrachloroethylene Pennsylvania RTK: Tetrachloroethylene Minnesota: Tetrachloroethylene Michigan critical material: Tetrachloroethylene Massachusetts RTK: Tetrachloroethylene Massachusetts spill list: Tetrachloroethylene New Jersey: Tetrachloroethylene New Jersey spill list: Tetrachloroethylene Louisiana spill reporting: Tetrachloroethylene California Director's List of Hazardous Substances: Tetrachloroethylene TSCA 8(b) inventory: Tetrachloroethylene TSCA 8(d) H and S data reporting: Tetrachloroethylene: Effective date: 6/1/87; Sunset date: 6/1/97 SARA 313 toxic chemical notification and release reporting: Tetrachloroethylene CERCLA: Hazardous substances.: Tetrachloroethylene: 100 lbs. (45.36 kg)

Other Regulations:
OSHA: Hazardous by definition of Hazard Communication Standard (29 CFR 1910.1200). EINECS: This product is on the European Inventory of Existing Commercial Chemical Substances.

Other Classifications:

WHMIS (Canada):
CLASS D-1B: Material causing immediate and serious toxic effects (TOXIC). CLASS D-2A: Material causing other toxic effects (VERY TOXIC).

DSCL (EEC):
R40- Possible risks of irreversible effects. R51/53- Toxic to aquatic organisms, may cause long-term adverse effects in the aquatic environment. S23- Do not breathe gas/fumes/vapour/spray S26- In case of contact with eyes, rinse immediately with plenty of water and seek medical advice. S37- Wear suitable gloves. S61- Avoid release to the environment. Refer to special instructions/Safety data sheets.

HMIS (U.S.A.):

Health Hazard: 2

Fire Hazard: 0

Reactivity: 0

Personal Protection: g

National Fire Protection Association (U.S.A.):

Health: 2

Flammability: 0

Reactivity: 0

Specific hazard:

Protective Equipment:
Gloves. Lab coat. Vapor respirator. Be sure to use an approved/certified respirator or equivalent. Wear appropriate respirator when ventilation is inadequate. Safety glasses.

Section 16: Other Information

References: Not available.

Other Special Considerations: Not available.

Created: 10/10/2005 08:29 PM

Last Updated: 11/01/2010 12:00 PM

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Safety data for trans-1,2-dichloroethylene

Glossary of terms on this data sheet.

The information on this web page is provided to help you to work safely, but it is intended to be an overview of hazards, not a replacement for a full Material Safety Data Sheet (MSDS). MSDS forms can be downloaded from the web sites of many chemical suppliers.

General

Synonyms: (E)-1,2-dichloroethene, trans-1,2-dichloroethene, trans-acetylene dichloride, 1,2-trans-dichloroethylene, 1,2-trans-dichloroethene, trans-1,2-DCE, Vertrel CCA
Use: Solvent, synthetic agent, constituent of perfumes
Molecular formula: $C_2H_2Cl_2$
CAS No: 156-60-5
EC No: 205-860-2

Physical data

Appearance: colourless liquid
Melting point: -50 C
Boiling point: 48 C
Vapour density:
Vapour pressure: 5.2 PSI at 20 C
Specific gravity: 1.257
Flash point: 6 C
Explosion limits: 9.7 - 12.8%
Autoignition temperature:

Stability

Incompatible with oxidizing agents, bases. Stable, but may decompose on exposure to air, moisture or light. Highly flammable.

Toxicology

Harmful if swallowed or inhaled, and in contact with skin. Narcotic. Irritant.

Toxicity data

(The meaning of any toxicological abbreviations which appear in this section is given here.)

ORL-RAT 1235 mg kg^{-1}

IHL-HMN TCLO 4800 mg m^{-3}/10m

IPR-RAT LD50 7536 mg kg^{-1}

ORL-MUS LD50 2122 mg kg^{-1}

Risk phrases

(The meaning of any risk phrases which appear in this section is given here.)
R11 R20 R21 R22 R36 R37 R38.

Transport information

(The meaning of any UN hazard codes which appear in this section is given here.)
UN No 1150. Hazard class 3.0. Packing group II

Personal protection

Safety glasses, adequate ventilation.

Safety phrases

(The meaning of any safety phrases which appear in this section is given here.)
S16 S26 S36.

[Return to Physical & Theoretical Chemistry Lab. Safety home page.]

This information was last updated on March 8, 2006. We have tried to make it as accurate and useful as possible, but can take no responsibility for its use, misuse, or accuracy. We have not verified this information, and cannot guarantee that it is up-to-date.

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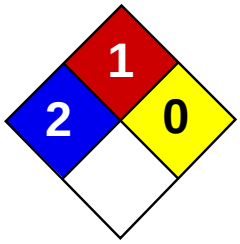

Material Safety Data Sheet
Trichloroethylene MSDS

Section 1: Chemical Product and Company Identification

Product Name: Trichloroethylene

Catalog Codes: SLT3310, SLT2590

CAS#: 79-01-6

RTECS: KX4560000

TSCA: TSCA 8(b) inventory: Trichloroethylene

CI#: Not available.

Synonym:

Chemical Formula: C2HCl3

Contact Information:

Sciencelab.com, Inc.
14025 Smith Rd.
Houston, Texas 77396

US Sales: **1-800-901-7247**
International Sales: **1-281-441-4400**

Order Online: ScienceLab.com

CHEMTREC (24HR Emergency Telephone), call:
1-800-424-9300

International CHEMTREC, call: 1-703-527-3887

For non-emergency assistance, call: 1-281-441-4400

Section 2: Composition and Information on Ingredients

Composition:

Name	CAS #	% by Weight
Trichloroethylene	79-01-6	100

Toxicological Data on Ingredients: Trichloroethylene: ORAL (LD50): Acute: 5650 mg/kg [Rat]. 2402 mg/kg [Mouse]. DERMAL (LD50): Acute: 20001 mg/kg [Rabbit].

Section 3: Hazards Identification

Potential Acute Health Effects: Hazardous in case of skin contact (irritant, permeator), of eye contact (irritant), of ingestion, of inhalation.

Potential Chronic Health Effects:
CARCINOGENIC EFFECTS: Classified + (PROVEN) by OSHA. Classified A5 (Not suspected for human.) by ACGIH. MUTAGENIC EFFECTS: Not available. TERATOGENIC EFFECTS: Not available. DEVELOPMENTAL TOXICITY: Not available. The substance is toxic to kidneys, the nervous system, liver, heart, upper respiratory tract. Repeated or prolonged exposure to the substance can produce target organs damage.

Section 4: First Aid Measures

Eye Contact:

Check for and remove any contact lenses. Immediately flush eyes with running water for at least 15 minutes, keeping eyelids open. Cold water may be used. Do not use an eye ointment. Seek medical attention.

Skin Contact:
After contact with skin, wash immediately with plenty of water. Gently and thoroughly wash the contaminated skin with running water and non-abrasive soap. Be particularly careful to clean folds, crevices, creases and groin. Cover the irritated skin with an emollient. If irritation persists, seek medical attention. Wash contaminated clothing before reusing.

Serious Skin Contact:
Wash with a disinfectant soap and cover the contaminated skin with an anti-bacterial cream. Seek medical attention.

Inhalation: Allow the victim to rest in a well ventilated area. Seek immediate medical attention.

Serious Inhalation:
Evacuate the victim to a safe area as soon as possible. Loosen tight clothing such as a collar, tie, belt or waistband. If breathing is difficult, administer oxygen. If the victim is not breathing, perform mouth-to-mouth resuscitation. Seek medical attention.

Ingestion:
Do not induce vomiting. Loosen tight clothing such as a collar, tie, belt or waistband. If the victim is not breathing, perform mouth-to-mouth resuscitation. Seek immediate medical attention.

Serious Ingestion: Not available.

Section 5: Fire and Explosion Data

Flammability of the Product: May be combustible at high temperature.

Auto-Ignition Temperature: 420°C (788°F)

Flash Points: Not available.

Flammable Limits: LOWER: 8% UPPER: 10.5%

Products of Combustion: These products are carbon oxides (CO, CO2), halogenated compounds.

Fire Hazards in Presence of Various Substances: Not available.

Explosion Hazards in Presence of Various Substances:
Risks of explosion of the product in presence of mechanical impact: Not available. Risks of explosion of the product in presence of static discharge: Not available.

Fire Fighting Media and Instructions:
SMALL FIRE: Use DRY chemical powder. LARGE FIRE: Use water spray, fog or foam. Do not use water jet.

Special Remarks on Fire Hazards: Not available.

Special Remarks on Explosion Hazards: Not available.

Section 6: Accidental Release Measures

Small Spill: Absorb with an inert material and put the spilled material in an appropriate waste disposal.

Large Spill:
Absorb with an inert material and put the spilled material in an appropriate waste disposal. Be careful that the product is not present at a concentration level above TLV. Check TLV on the MSDS and with local authorities.

Section 7: Handling and Storage

Precautions:
Keep locked up Keep away from heat. Keep away from sources of ignition. Empty containers pose a fire risk, evaporate the residue under a fume hood. Ground all equipment containing material. Do not ingest. Do not breathe gas/fumes/ vapour/

spray. Wear suitable protective clothing In case of insufficient ventilation, wear suitable respiratory equipment If ingested, seek medical advice immediately and show the container or the label. Avoid contact with skin and eyes

Storage:
Keep container dry. Keep in a cool place. Ground all equipment containing material. Carcinogenic, teratogenic or mutagenic materials should be stored in a separate locked safety storage cabinet or room.

Section 8: Exposure Controls/Personal Protection

Engineering Controls:
Provide exhaust ventilation or other engineering controls to keep the airborne concentrations of vapors below their respective threshold limit value. Ensure that eyewash stations and safety showers are proximal to the work-station location.

Personal Protection:
Splash goggles. Lab coat. Vapor respirator. Be sure to use an approved/certified respirator or equivalent. Gloves.

Personal Protection in Case of a Large Spill:
Splash goggles. Full suit. Vapor respirator. Boots. Gloves. A self contained breathing apparatus should be used to avoid inhalation of the product. Suggested protective clothing might not be sufficient; consult a specialist BEFORE handling this product.

Exposure Limits:
TWA: 50 STEL: 200 (ppm) from ACGIH (TLV) TWA: 269 STEL: 1070 (mg/m3) from ACGIH Consult local authorities for acceptable exposure limits.

Section 9: Physical and Chemical Properties

Physical state and appearance: Liquid.

Odor: Not available.

Taste: Not available.

Molecular Weight: 131.39 g/mole

Color: Clear Colorless.

pH (1% soln/water): Not available.

Boiling Point: 86.7°C (188.1°F)

Melting Point: -87.1°C (-124.8°F)

Critical Temperature: Not available.

Specific Gravity: 1.4649 (Water = 1)

Vapor Pressure: 58 mm of Hg (@ 20°C)

Vapor Density: 4.53 (Air = 1)

Volatility: Not available.

Odor Threshold: 20 ppm

Water/Oil Dist. Coeff.: The product is equally soluble in oil and water; log(oil/water) = 0

Ionicity (in Water): Not available.

Dispersion Properties: See solubility in water, methanol, diethyl ether, acetone.

Solubility:
Easily soluble in methanol, diethyl ether, acetone. Very slightly soluble in cold water.

Section 10: Stability and Reactivity Data

Stability: The product is stable.

Instability Temperature: Not available.

Conditions of Instability: Not available.

Incompatibility with various substances: Not available.

Corrosivity:
Extremely corrosive in presence of aluminum. Non-corrosive in presence of glass.

Special Remarks on Reactivity: Not available.

Special Remarks on Corrosivity: Not available.

Polymerization: No.

Section 11: Toxicological Information

Routes of Entry: Dermal contact. Eye contact. Inhalation. Ingestion.

Toxicity to Animals:
Acute oral toxicity (LD50): 2402 mg/kg [Mouse]. Acute dermal toxicity (LD50): 20001 mg/kg [Rabbit].

Chronic Effects on Humans:
CARCINOGENIC EFFECTS: Classified + (PROVEN) by OSHA. Classified A5 (Not suspected for human.) by ACGIH. The substance is toxic to kidneys, the nervous system, liver, heart, upper respiratory tract.

Other Toxic Effects on Humans: Hazardous in case of skin contact (irritant, permeator), of ingestion, of inhalation.

Special Remarks on Toxicity to Animals: Not available.

Special Remarks on Chronic Effects on Humans: Passes through the placental barrier in human. Detected in maternal milk in human.

Special Remarks on other Toxic Effects on Humans: Not available.

Section 12: Ecological Information

Ecotoxicity: Not available.

BOD5 and COD: Not available.

Products of Biodegradation:
Possibly hazardous short term degradation products are not likely. However, long term degradation products may arise.

Toxicity of the Products of Biodegradation: The products of degradation are more toxic.

Special Remarks on the Products of Biodegradation: Not available.

Section 13: Disposal Considerations

Waste Disposal:

Section 14: Transport Information

DOT Classification: CLASS 6.1: Poisonous material.

Identification: : Trichloroethylene : UN1710 PG: III

Special Provisions for Transport: Not available.

Section 15: Other Regulatory Information

Federal and State Regulations:
California prop. 65: This product contains the following ingredients for which the State of California has found to cause cancer, birth defects or other reproductive harm, which would require a warning under the statute: Trichloroethylene California prop. 65: This product contains the following ingredients for which the State of California has found to cause cancer which would require a warning under the statute: Trichloroethylene Pennsylvania RTK: Trichloroethylene Florida: Trichloroethylene Minnesota: Trichloroethylene Massachusetts RTK: Trichloroethylene New Jersey: Trichloroethylene TSCA 8(b) inventory: Trichloroethylene CERCLA: Hazardous substances.: Trichloroethylene

Other Regulations: OSHA: Hazardous by definition of Hazard Communication Standard (29 CFR 1910.1200).

Other Classifications:

WHMIS (Canada):
CLASS D-1B: Material causing immediate and serious toxic effects (TOXIC). CLASS D-2B: Material causing other toxic effects (TOXIC).

DSCL (EEC):
R36/38- Irritating to eyes and skin. R45- May cause cancer.

HMIS (U.S.A.):

Health Hazard: 2

Fire Hazard: 1

Reactivity: 0

Personal Protection: h

National Fire Protection Association (U.S.A.):

Health: 2

Flammability: 1

Reactivity: 0

Specific hazard:

Protective Equipment:
Gloves. Lab coat. Vapor respirator. Be sure to use an approved/certified respirator or equivalent. Wear appropriate respirator when ventilation is inadequate. Splash goggles.

Section 16: Other Information

References: Not available.

Other Special Considerations: Not available.

Created: 10/10/2005 08:54 PM

Last Updated: 11/01/2010 12:00 PM

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MATERIAL SAFETY DATA SHEET

1. CHEMICAL PRODUCT AND COMPANY IDENTIFICATION

MATHESON TRI-GAS, INC.
150 Allen Road Suite 302
Basking Ridge, New Jersey 07920
Information: 1-800-416-2505

Emergency Contact:
CHEMTREC 1-800-424-9300
Calls Originating Outside the US:
703-527-3887 (Collect Calls Accepted)

SUBSTANCE: VINYL CHLORIDE

TRADE NAMES/SYNONYMS:
MTG MSDS 97; 1-CHLOROETHYLENE; 1-CHLOROETHENE; CHLOROETHYLENE; CHLOROETHENE; CHLORETHENE; CHLORETHYLENE; ETHYLENE MONOCHLORIDE; MONOCHLOROETHYLENE; MONOCHLORO ETHENE; MONOCHLOROETHENE; VINYL CHLORIDE MONOMER; VINYL CHLORIDE, INHIBITED; VINYL C MONOMER; RCRA U043; UN 1086; C2H3Cl; MAT24940; RTECS KU9625000

CHEMICAL FAMILY: halogenated, aliphatic

CREATION DATE: Jan 24 1989
REVISION DATE: Dec 11 2008

2. COMPOSITION, INFORMATION ON INGREDIENTS

COMPONENT: VINYL CHLORIDE
CAS NUMBER: 75-01-4
PERCENTAGE: >99.9

COMPONENT: PHENOL
CAS NUMBER: 108-95-2
PERCENTAGE: <0.1

COMPONENT: INHIBITORS
CAS NUMBER: Not assigned.
PERCENTAGE: <0.1

3. HAZARDS IDENTIFICATION

NFPA RATINGS (SCALE 0-4): HEALTH=2 FIRE=4 REACTIVITY=1

EMERGENCY OVERVIEW:
COLOR: colorless
PHYSICAL FORM: gas
ODOR: faint odor, sweet odor
MAJOR HEALTH HAZARDS: harmful if swallowed, skin irritation, eye irritation, central nervous system depression, cancer hazard (in humans)
PHYSICAL HAZARDS: Flammable gas. May cause flash fire. May polymerize. Containers may rupture or explode.

POTENTIAL HEALTH EFFECTS:
INHALATION:
SHORT TERM EXPOSURE: irritation, nausea, difficulty breathing, irregular heartbeat, headache, drowsiness, dizziness, disorientation, joint pain, loss of coordination, hearing loss, lung congestion
LONG TERM EXPOSURE: impotence, bluish skin color, blood disorders, liver damage, cancer
SKIN CONTACT:
SHORT TERM EXPOSURE: irritation, blisters
LONG TERM EXPOSURE: irritation, blisters
EYE CONTACT:
SHORT TERM EXPOSURE: irritation, eye damage
LONG TERM EXPOSURE: irritation, eye damage
INGESTION:
SHORT TERM EXPOSURE: frostbite
LONG TERM EXPOSURE: cancer

4. FIRST AID MEASURES

INHALATION: If adverse effects occur, remove to uncontaminated area. Give artificial respiration if not breathing. If breathing is difficult, oxygen should be administered by qualified personnel. Get immediate medical attention.

SKIN CONTACT: If frostbite or freezing occur, immediately flush with plenty of lukewarm water (105-115 F; 41-46 C). DO NOT USE HOT WATER. If warm water is not available, gently wrap affected parts in blankets. Get immediate medical attention.

EYE CONTACT: Wash eyes immediately with large amounts of water, occasionally lifting upper and lower lids, until no evidence of chemical remains. Get medical attention immediately.

INGESTION: If a large amount is swallowed, get medical attention.

NOTE TO PHYSICIAN: For inhalation, consider oxygen.

5. FIRE FIGHTING MEASURES

FIRE AND EXPLOSION HAZARDS: Severe fire hazard. Severe explosion hazard. The vapor is heavier than air. Vapors or gases may ignite at distant ignition sources and flash back. Vapor/air mixtures are explosive. Electrostatic discharges may be generated by flow or agitation resulting in ignition or explosion.

EXTINGUISHING MEDIA: carbon dioxide, regular dry chemical

Large fires: Use regular foam or flood with fine water spray.

FIRE FIGHTING: Move container from fire area if it can be done without risk. For fires in cargo or storage area: Cool containers with water from unmanned hose holder or monitor nozzles until well after fire is out. If this is impossible then take the following precautions: Keep unnecessary people away, isolate hazard area and deny entry. Let the fire burn. Withdraw immediately in case of rising sound from venting safety device or any discoloration of tanks due to fire. For tank, rail car or tank truck: Stop leak if possible without personal risk. Let burn unless leak can be stopped immediately. For smaller tanks or cylinders, extinguish and isolate from other flammables. Evacuation radius: 800 meters (1/2 mile). Do not attempt to extinguish fire unless flow of material can be stopped first. Flood with fine water spray. Cool containers with water spray until well after the fire is out. Apply water from a protected location or from a safe distance. Avoid inhalation of material or combustion by-products. Stay upwind and keep out of low areas. Evacuate if fire gets out of control or containers are directly exposed to fire. Evacuation radius: 500 meters (1/3 mile). Consider downwind evacuation if material is leaking.

FLASH POINT: -108 F (-78 C) (CC)
LOWER FLAMMABLE LIMIT: 3.6%
UPPER FLAMMABLE LIMIT: 33%
AUTOIGNITION: 882 F (472 C)

6. ACCIDENTAL RELEASE MEASURES

WATER RELEASE:
Subject to California Safe Drinking Water and Toxic Enforcement Act of 1986 (Proposition 65). Keep out of water supplies and sewers.

OCCUPATIONAL RELEASE:
Avoid heat, flames, sparks and other sources of ignition. Stop leak if possible without personal risk. Reduce vapors with water spray. Keep unnecessary people away, isolate hazard area and deny entry. Remove sources of ignition. Ventilate closed spaces before entering. Notify Local Emergency Planning Committee and State Emergency Response Commission for release greater than or equal to RQ (U.S. SARA Section 304). If release occurs in the U.S. and is reportable under CERCLA Section 103, notify the National Response Center at (800)424-8802 (USA) or (202)426-2675 (USA).

7. HANDLING AND STORAGE

STORAGE: Store and handle in accordance with all current regulations and standards. Protect from physical damage. Store outside or in a detached building. Inside storage: Store in a cool, dry place. Store in a

well-ventilated area. Avoid heat, flames, sparks and other sources of ignition. Grounding and bonding required. Subject to storage regulations: U.S. OSHA 29 CFR 1910.101. See original container for storage recommendations. Keep separated from incompatible substances.

8. EXPOSURE CONTROLS, PERSONAL PROTECTION

EXPOSURE LIMITS:
VINYL CHLORIDE:
1.0 ppm OSHA TWA
5 ppm OSHA STEL 15 minute(s)
0.5 ppm OSHA action level 8 hour(s)
1 ppm ACGIH TWA
NIOSH TWA (lowest feasible concentration)

VENTILATION: Ventilation equipment should be explosion-resistant if explosive concentrations of material are present. Provide local exhaust or process enclosure ventilation system. Ensure compliance with applicable exposure limits.

EYE PROTECTION: Wear splash resistant safety goggles with a faceshield. Provide an emergency eye wash fountain and quick drench shower in the immediate work area.

CLOTHING: Wear appropriate chemical resistant clothing.

GLOVES: For the gas: Wear appropriate chemical resistant gloves. For the liquid: Wear insulated gloves.
OSHA REGULATED SUBSTANCES: U.S. OSHA 29 CFR 1910.1017.

RESPIRATOR: The following respirators and maximum use concentrations are drawn from NIOSH and/or OSHA.
OSHA Standard:
Respirator selection should comply with 29 CFR 1910.134, 29 CFR 1910.1017, and the final rule published in the Federal Register on August 24, 2006.
NIOSH Recommendations:
At any detectable concentration -
Any self-contained breathing apparatus that has a full facepiece and is operated in a pressure-demand or other positive-pressure mode.
Any supplied-air respirator with a full facepiece that is operated in a pressure-demand or other positive-pressure mode in combination with an auxiliary self-contained breathing apparatus operated in pressure-demand or other positive-pressure mode.
Escape -
Any air-purifying full-facepiece respirator (gas mask) with a chin-style, front-mounted or back-mounted canister providing protection against the compound of concern.
Any appropriate escape-type, self-contained breathing apparatus.

9. PHYSICAL AND CHEMICAL PROPERTIES

PHYSICAL STATE: gas
COLOR: colorless
ODOR: faint odor, sweet odor
MOLECULAR WEIGHT: 62.50
MOLECULAR FORMULA: C-H2-C-H-Cl
BOILING POINT: 9 F (-13 C)
FREEZING POINT: -245 F (-154 C)
VAPOR PRESSURE: 2515.6 mmHg @ 21.1 C
VAPOR DENSITY (air=1): 2.2
SPECIFIC GRAVITY (water=1): 0.9106
WATER SOLUBILITY: 0.25%
PH: Not applicable
VOLATILITY: Not applicable
ODOR THRESHOLD: 260 ppm
EVAPORATION RATE: Not applicable
VISCOSITY: 0.01072 cP @ 20 C
COEFFICIENT OF WATER/OIL DISTRIBUTION: Not applicable
SOLVENT SOLUBILITY:
Soluble: alcohol, ether, carbon tetrachloride, benzene

10. STABILITY AND REACTIVITY

REACTIVITY: May polymerize. Avoid contact with light or storage and use above room temperature.

CONDITIONS TO AVOID: Avoid heat, flames, sparks and other sources of ignition. Containers may rupture or explode if exposed to heat.

INCOMPATIBILITIES: metal carbide, metals, oxidizing materials, peroxides

HAZARDOUS DECOMPOSITION:
Thermal decomposition products: halogenated compounds, oxides of carbon, phosgene

POLYMERIZATION: May polymerize. Avoid contact with heat, light, air, water or incompatible materials. Closed containers may rupture violently.

11. TOXICOLOGICAL INFORMATION

VINYL CHLORIDE:
TOXICITY DATA: 18 pph/15 minute(s) inhalation-rat LC50; 500 mg/kg oral-rat LD50
CARCINOGEN STATUS: OSHA: Carcinogen; NTP: Known Human Carcinogen; IARC: Human Sufficient Evidence, Animal Sufficient Evidence, Group 1; ACGIH: A1 -Confirmed Human Carcinogen;

EC: Category 1
LOCAL EFFECTS:
Irritant: skin, eye
ACUTE TOXICITY LEVEL:
Toxic: ingestion
Relatively Non-toxic: inhalation
TARGET ORGANS: central nervous system
TUMORIGENIC DATA: Available.
MUTAGENIC DATA: Available.
REPRODUCTIVE EFFECTS DATA: Available.
ADDITIONAL DATA: Stimulants such as epinephrine may induce ventricular fibrillation. May cause birth defects.

12. ECOLOGICAL INFORMATION

ECOTOXICITY DATA:
FISH TOXICITY: 388000 ug/L 10 month(s) LETH (Mortality) Northern pike (Esox lucius)

INVERTEBRATE TOXICITY: 41.74 ug/L 72 day(s) (Residue) Mosquito (Culex pipiens quinquefasciata)

ALGAL TOXICITY: 41.74 ug/L 72 day(s) (Residue) Green algae (Oedogonium cardiacum)

13. DISPOSAL CONSIDERATIONS

Dispose in accordance with all applicable regulations. Hazardous Waste Number(s): D043. Dispose of in accordance with U.S. EPA 40 CFR 262 for concentrations at or above the Regulatory level. Regulatory level- 0.2 mg/L. U043.

14. TRANSPORT INFORMATION

U.S. DOT 49 CFR 172.101:
PROPER SHIPPING NAME: Vinyl chloride, stabilized
ID NUMBER: UN1086
HAZARD CLASS OR DIVISION: 2.1
LABELING REQUIREMENTS: 2.1
QUANTITY LIMITATIONS:
PASSENGER AIRCRAFT OR RAILCAR: Forbidden
CARGO AIRCRAFT ONLY: 150 kg

CANADIAN TRANSPORTATION OF DANGEROUS GOODS:
SHIPPING NAME: Vinyl chloride, stabilized
UN NUMBER: UN1086
CLASS: 2.1

15. REGULATORY INFORMATION

U.S. REGULATIONS:
CERCLA SECTIONS 102a/103 HAZARDOUS SUBSTANCES (40 CFR 302.4):
Vinyl chloride: 1 LBS RQ
PHENOL: 1000 LBS RQ

SARA TITLE III SECTION 302 EXTREMELY HAZARDOUS SUBSTANCES (40 CFR 355 Subpart B): Not regulated.

SARA TITLE III SECTION 304 EXTREMELY HAZARDOUS SUBSTANCES (40 CFR 355 Subpart C): Not regulated.

SARA TITLE III SARA SECTIONS 311/312 HAZARDOUS CATEGORIES (40 CFR 370 Subparts B and C):
ACUTE: Yes
CHRONIC: Yes
FIRE: Yes
REACTIVE: Yes
SUDDEN RELEASE: Yes

SARA TITLE III SECTION 313 (40 CFR 372.65):
Vinyl chloride

OSHA PROCESS SAFETY (29 CFR 1910.119): Not regulated.

STATE REGULATIONS:
California Proposition 65:
Known to the state of California to cause the following:
Vinyl chloride
Cancer (Feb 27, 1987)

CANADIAN REGULATIONS:
WHMIS CLASSIFICATION: ABD2

NATIONAL INVENTORY STATUS:
U.S. INVENTORY (TSCA): Listed on inventory.

TSCA 12(b) EXPORT NOTIFICATION: Not listed.

CANADA INVENTORY (DSL/NDSL): Not determined.

16. OTHER INFORMATION

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